

AUTHOR SEARCH

L18 ANSWER 1 OF 3 HCAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 2005:1106865 HCAPLUS Full-text

DOCUMENT NUMBER: 143:387237

TITLE: Process for the preparation of 5 α -pregnane
derivative via reduction of carbon-carbon double bond
in pregn-4-en-3-one compound

INVENTOR(S): Sugioka, Takashi; Ohzono, Shigeo;
Koyakumaru, Kenichi; Nakagawa, Naoshi

PATENT ASSIGNEE(S): Kuraray Co., Ltd., Japan

SOURCE: PCT Int. Appl., 27 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent

LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

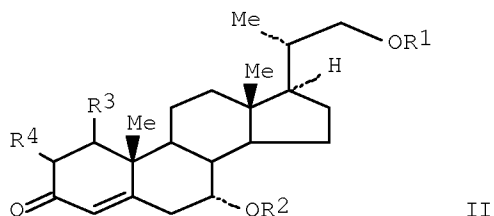
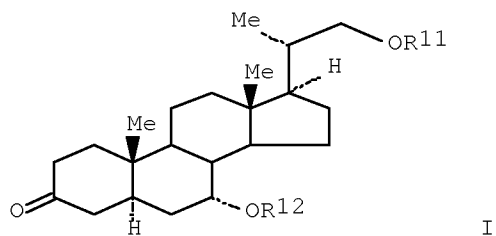
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2005095434	A1	20051013	WO 2005-JP6828	20050331
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SM, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW				
RW: BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
EP 1743902	A1	20070117	EP 2005-728917	20050331
R: AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LI, LT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR				
CN 1934124	A	20070321	CN 2005-80009530	20050331
US 20070149494	A1	20070628	US 2006-594164	20060926
IN 2006CN03994	A	20070706	IN 2006-CN3994	20061031
PRIORITY APPLN. INFO.:			JP 2004-108419	A 20040331
			WO 2005-JP6828	W 20050331

OTHER SOURCE(S): MARPAT 143:387237

ED Entered STN: 14 Oct 2005

GI



AB A process for the preparation of compound I [R11, R12 = H, protecting group of hydroxy], characterized by reduction a pregnane derivative II [R1 = protecting group of hydroxy; R2 = H, protecting group of hydroxy; R3 and R4 represent hydrogen atoms or combine together to form a bond.] with alkali metals or alkaline earth metals in the presence of a proton donor, amine and/or ammonia, was disclosed. For example, to a solution of (20S)-21-tert-butylldimethylsilyloxy-7 α -hydroxy-20-methylpregn-4-en-3-one (5.00 g) and tert-butanol (1.78 g) in THF (100 mL) was added liquid ammonia (100 mL) at -50 °C. Then, Li metal (0.17 g) was added, while maintaining the reaction temperature between -50 and -40 °C, the reaction was stirred -40 °C for 3 h. The resulting reaction was treated with ammonium sulfate (1.59 g) followed by removal of ammonia, aqueous work-up and silica-gel purification to give (20S)-21-tert-butylldimethylsilyloxy-7 α -hydroxy-20-methyl-5 α -pregn-3-one in 96% yield. Of note compds. I are useful synthetic intermediates for the preparation of squalamine.

REFERENCE COUNT: 6 THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L18 ANSWER 2 OF 3 HCAPLUS COPYRIGHT 2009 ACS on STN
 ACCESSION NUMBER: 2005:1103797 HCAPLUS [Full-text](#)
 DOCUMENT NUMBER: 143:387233
 TITLE: Method for producing 5 α -pregnane derivative
 INVENTOR(S): Koyakumaru, Kenichi; Sugioka, Takashi; Ohzono, Shigeo; Nakagawa, Naoshi
 PATENT ASSIGNEE(S): Kuraray Co., Ltd., Japan
 SOURCE: PCT Int. Appl., 33 pp.
 CODEN: PIXXD2
 DOCUMENT TYPE: Patent
 LANGUAGE: Japanese
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
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WO 2005095431	A1	20051013	WO 2005-JP6818	20050331
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH,				

Serial No.:10/594,163

CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SM, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW
 RW: BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

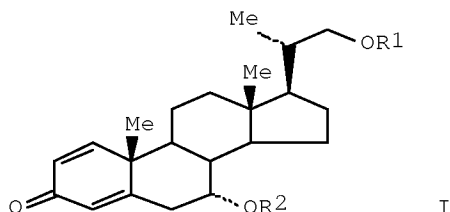
CN 1938330 A 20070328 CN 2005-80010110 20050331
 EP 1767540 A1 20070328 EP 2005-728889 20050331

R: AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LI, LT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR

US 20070197490 A1 20070823 US 2006-594163 20060926
 IN 2006CN03996 A 20070706 IN 2006-CN3996 20061031

PRIORITY APPLN. INFO.: JP 2004-108434 A 20040331
 WO 2005-JP6818 W 20050331

OTHER SOURCE(S): MARPAT 143:387233
 ED Entered STN: 14 Oct 2005
 GI



AB 5 α -Pregna-3-one derivs. and 5 α -pregna-1-en-3-one derivs. are prepared by reacting a pregnane derivative represented by the general formula I [R1 = OH-protecting group; R2 = H, OH-protecting group] with a metal selected from alkali metals and alkaline earth metals in the presence of a proton donor, an amine and/or ammonia. The title compds. are intermediates for squalamine. Thus, (20S)-21-tert-butyltrimethylsilyloxy-7 α -hydroxy-20-methyl-5 α -pregna-3-one and (20S)-21-tert-butyltrimethylsilyloxy-7 α -hydroxy-20-methyl-5 α -pregna-1-en-3-one were prepared by the title method.

REFERENCE COUNT: 6 THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L18 ANSWER 3 OF 3 HCAPLUS COPYRIGHT 2009 ACS on STN
 ACCESSION NUMBER: 2005:1103727 HCAPLUS Full-text
 DOCUMENT NUMBER: 143:386684

TITLE: Process for the preparation of cyclopropane monoacetal derivative and intermediate therefor

INVENTOR(S): Koyakumaru, Kenichi; Ueyama, Shingo; Ujita, Katsuji; Hayashibara, Tatsuhiko; Nakagawa, Naoshi; Akiba, Toshifumi; Saito, Tatsuru

PATENT ASSIGNEE(S): Kuraray Co., Ltd., Japan; Daiichi Pharmaceutical Co., Ltd.

SOURCE: PCT Int. Appl., 24 pp.

Serial No.:10/594,163

CODEN: PIXXD2

DOCUMENT TYPE:

Patent

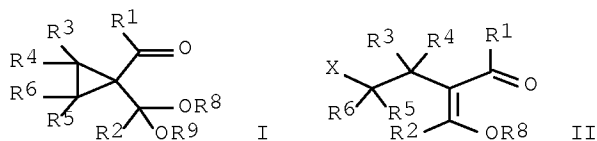
LANGUAGE:

Japanese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2005095318	A1	20051013	WO 2005-JP6407	20050325
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SM, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW RW: BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
EP 1731495	A1	20061213	EP 2005-727496	20050325
R: AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LI, LT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR				
US 20070191643	A1	20070816	US 2006-593129	20060918
US 7358400	B2	20080415		
PRIORITY APPLN. INFO.:			JP 2004-104862	A 20040331
			WO 2005-JP6407	W 20050325
OTHER SOURCE(S): MARPAT 143:386684				
ED Entered STN: 14 Oct 2005				
GI				



AB A process for industrially advantageously and easily producing a cyclopropane monoacetal derivative represented by the general formula I [R1-R6 = independently H, (un)substituted saturated hydrocarbon, aryl, alkenyl or aralkyl; R8, R9 = independently (un)substituted saturated hydrocarbon, aryl or aralkyl] through a small number of steps, characterized by reacting a halogenated unsatd. carbonyl compound represented by the general formula II [R1-R6 and R8 are defined as above; X = halo] with an alcoholate. For example, reaction of tri-Et orthoformate with 2,3-dihydrofuran, and followed by chlorination with thionyl chloride, gave 4-chloro-2-ethoxymethylidenebutanal (III). Reaction of III with sodium ethoxide provided 1-(diethoxymethyl)cyclopropanecarbaldehyde.

REFERENCE COUNT: 6 THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

STRUCTURE SEARCH

L3 ANSWER 1 OF 1 CASREACT COPYRIGHT 2009 ACS on STN
 AN 136:247742 CASREACT Full-text
 TI Process for the preparation of pregnane derivatives
 IN Nakazawa, Makoto
 PA Kuraray Co., Ltd., Japan
 SO PCT Int. Appl., 52 pp.
 CODEN: PIXXD2
 DT Patent
 LA Japanese
 IC ICM C07J005-00
 CC 32-5 (Steroids)

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 2002020552	A1	20020314	WO 2001-JP7639	20010904
	W: CA, CN, HU, IN, MX, US				
	RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR				
	JP 2002201199	A	20020716	JP 2001-261586	20010830
	CA 2416850	A1	20030120	CA 2001-2416850	20010904
	EP 1325928	A1	20030709	EP 2001-961343	20010904
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, FI, CY, TR				
	US 20030181742	A1	20030925	US 2003-363405	20030304
PRAI	JP 2000-273387		20000908		
	WO 2001-JP7639		20010904		
OS	MARPAT 136:247742				
GI					

* STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY - AVAILABLE VIA OFFLINE PRINT *

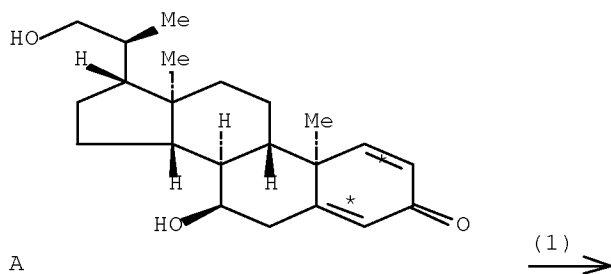
AB A process for the preparation of pregnane derivs. [I; R1 = H; R2 = protecting group; R3, R4 independently = alkyl, aryl, alkenyl, alkynyl; R3R4 = alkylene] which comprises reacting a compound II with an alkali metal or an alkaline earth metal in the presence of ammonia or an amine to obtain a compound III (R1 = H; R2 = H), protecting the hydroxyl groups of the compound III to obtain a compound III (R1 = protecting group; R2 = protecting group) protecting the compound III (R1 = protecting group; R2 = protecting group) at the 3-position to obtain a compound I (R1 = protecting group; R2 = protecting group; R3, R4 independently = alkyl, aryl, alkenyl, alkynyl; R3R4 = alkylene), and subjecting the compound I (R1 = protecting group; R2 = protecting group; R3, R4 independently = alkyl, aryl, alkenyl, alkynyl; R3R4 = alkylene) to solvolysis to obtain a compound I (R1 = H; R2 = protecting group; R3, R4 independently = alkyl, aryl, alkenyl, alkynyl; R3R4 = alkylene) and compound III (R1 = H; R2 = H). Thus, the title compound I (R1 = H; R2 = C6H5CO; R3R4 = CH2CH2) was prepared effectively from (20S)-7- α -hydroxy-3-oxo-pregna-1,4-diene-20-carboxaldehyde and C6H5COCl via hydrogenation and ethylene glycol and C6H5COCl O-protection and was useful as intermediate for squalamine preparation

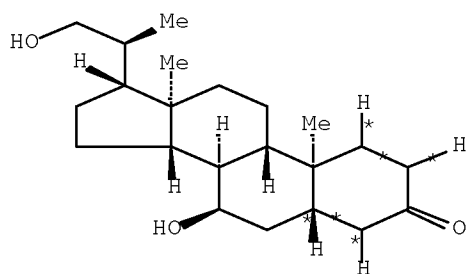
ST pregnane prepn redn hydrogenation solvolysis
 IT Hydrogenation
 Reduction
 Solvolysis

(process for the preparation of pregnane derivs.)
 IT 5132-07-0 7681-52-9
 RL: RGT (Reagent); RACT (Reactant or reagent)
 (compound for comparison in process for the preparation of pregnane derivs.)
 IT 122197-36-8
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (process for the preparation of pregnane derivs.)
 IT 208833-68-5P 296768-82-6P 403854-15-9P 403854-16-0P 403854-17-1P
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
 (process for the preparation of pregnane derivs.)
 IT 6192-52-5, p-Toluenesulfonic acid monohydrate
 RL: RGT (Reagent); RACT (Reactant or reagent)
 (process for the preparation of pregnane derivs.)
 IT 208254-12-0P 301695-48-7P
 RL: SPN (Synthetic preparation); PREP (Preparation)
 (process for the preparation of pregnane derivs.)
 IT 107-21-1, Ethylene glycol, reactions
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (protecting process for the preparation of pregnane derivs.)
 RE.CNT 12 THERE ARE 12 CITED REFERENCES AVAILABLE FOR THIS RECORD
 RE

- (1) Anon; Bioorg Med Chem 2000, V8(8), P2059
- (2) Anon; Chem Pharm Bull 1993, V41(4), P763
- (3) Anon; Organic Letters 2000, V2(19), P2921
- (4) Hoffmann La Roche F Und Co A G; EP 18515 A2 1980 CAPLUS
- (5) Hoffmann La Roche F Und Co A G; US 4230625 A 1980 CAPLUS
- (6) Hoffmann La Roche F Und Co A G; US 4301246 A 1980 CAPLUS
- (7) Hoffmann La Roche F Und Co A G; JP 568399 A 1980
- (8) Magainin Pharm Inc; JP 08507527 A 1994
- (9) Magainin Pharm Inc; US 5637691 A 1994 CAPLUS
- (10) Magainin Pharm Inc; EP 688333 A1 1994 CAPLUS
- (11) Magainin Pharm Inc; WO 9420520 A1 1994 CAPLUS
- (12) Magainin Pharm Inc; AU 9463974 A 1994 CAPLUS

RX(1) OF 35 ...A ==> B...





B
YIELD 65%

RX(1) RCT A 296768-82-6

STAGE(1)

SOL 109-99-9 THF

STAGE(2)

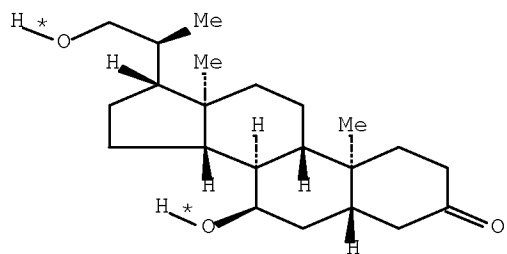
RGT C 7664-41-7 NH₃, D 7439-93-2 Li

SOL 7732-18-5 Water

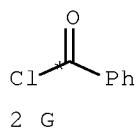
PRO B 301695-48-7

NTE -78°, under nitrogen

RX(2) OF 35 ...B + 2 G ==> H...

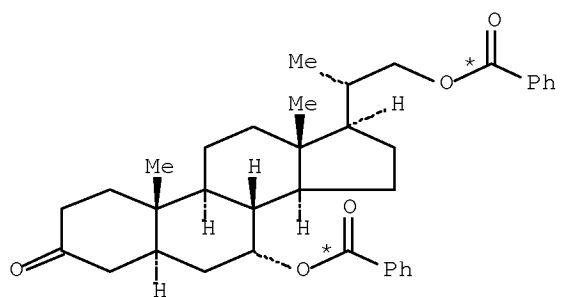


B



2 G

(2) →



^H
YIELD 90%

RX(2) RCT B 301695-48-7

STAGE(1)

RGT I 1122-58-3 4-DMAP, J 110-86-1 Pyridine
SOL 75-09-2 CH₂Cl₂

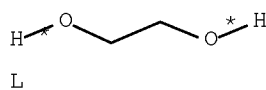
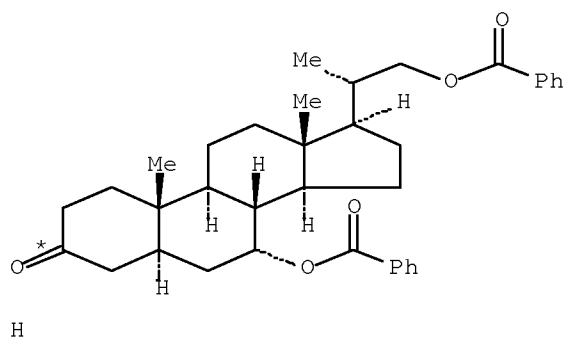
STAGE(2)

RCT G 98-88-4

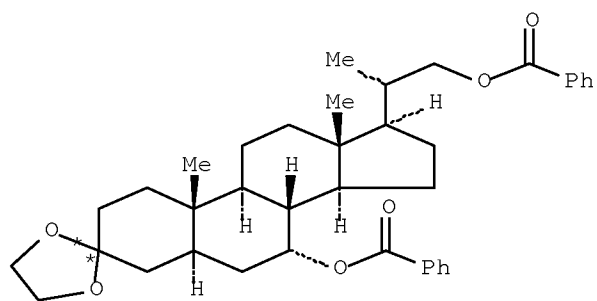
PRO H 403854-15-9

NTE 0°, under nitrogen in first step, room temp. for 12 h in
second step

RX(3) OF 35 ...H + L ==> M...



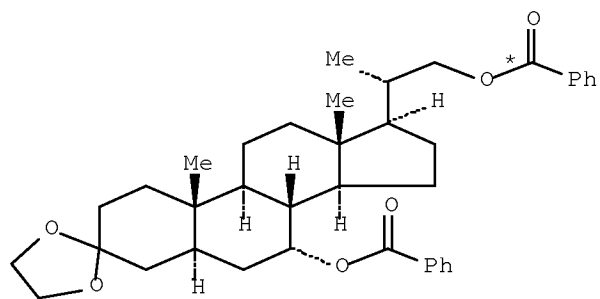
(3)
→



M
YIELD 97%

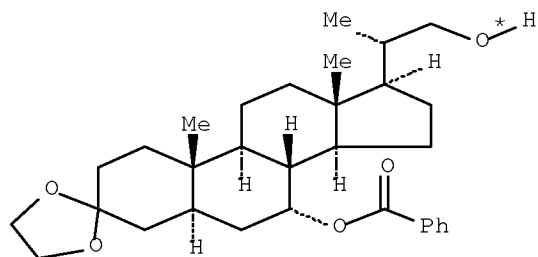
RX(3) RCT H 403854-15-9, L 107-21-1
 RGT N 104-15-4 TsOH
 PRO M 403854-16-0
 SOL 108-88-3 PhMe
 NTE reflux, 2 h

RX(4) OF 35 ...M ==> P...



M

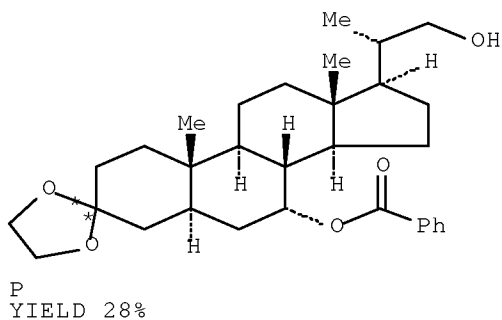
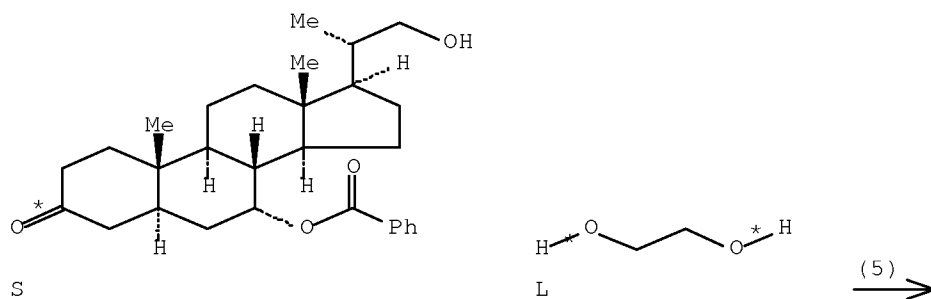
(4)
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P
YIELD 86%

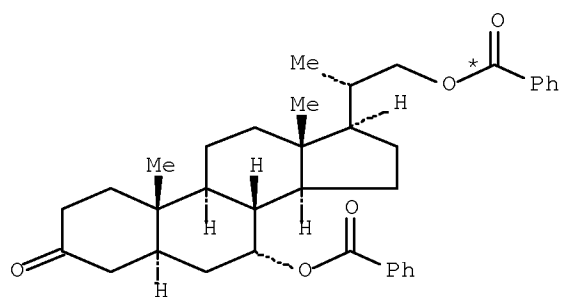
RX(4) RCT M 403854-16-0
 RGT Q 1310-73-2 NaOH
 PRO P 208833-68-5
 SOL 67-56-1 MeOH, 109-99-9 THF
 NTE room temp., 11 h

RX(5) OF 35 ...S + L ==> P...



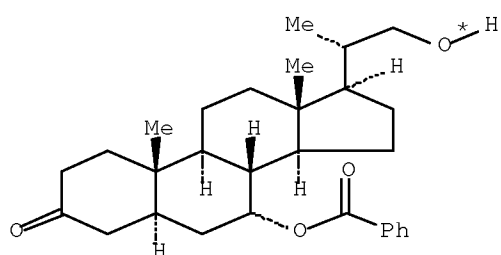
RX(5) RCT S 403854-17-1, L 107-21-1
 RGT T 6192-52-5 p-MeC6H4SO3H.H2O
 PRO P 208833-68-5
 SOL 108-88-3 PhMe
 NTE reflux, 40 h

RX(6) OF 35 ...H ==> S...



H

(6) →



S
YIELD 69%

RX(6) RCT H 403854-15-9

STAGE(1)

SOL 109-99-9 THF

STAGE(2)

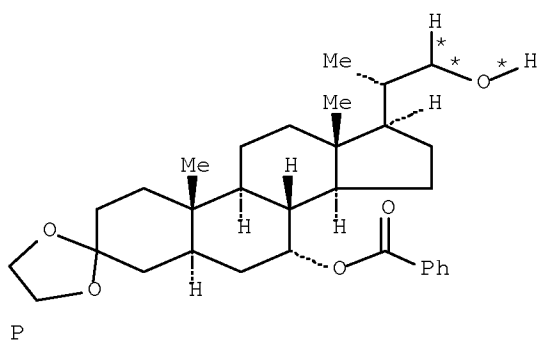
RGT U 497-19-8 Na₂CO₃, Q 1310-73-2 NaOH

SOL 7732-18-5 Water, 67-56-1 MeOH

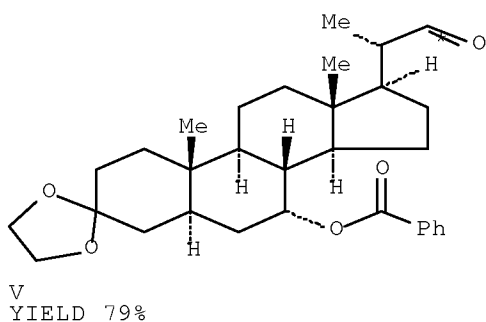
PRO S 403854-17-1

NTE 40°, 4 h

RX(7) OF 35 ...P ==> V



(7) →



RX(7) RCT P 208833-68-5

STAGE(1)

RGT W 5132-07-0 Piperidine, 2,2,6,6-tetramethyl-, 1-oxide
SOL 75-09-2 CH₂Cl₂

STAGE(2)

RGT X 7758-02-3 KBr
SOL 7732-18-5 Water

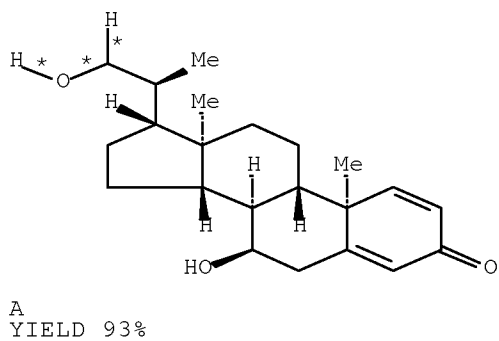
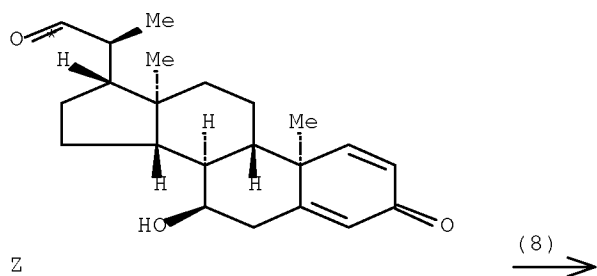
STAGE(3)

RGT Y 7681-52-9 NaOCl, U 497-19-8 Na₂CO₃
SOL 7732-18-5 Water

PRO V 208254-12-0

NTE 0°, 2 h

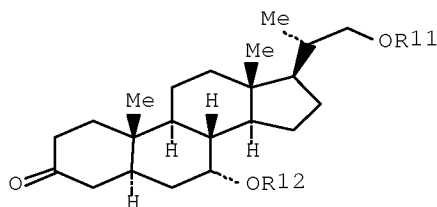
RX(8) OF 35 Z ==> A...



RX(8)	RCT	Z	122197-36-8
	RGT	AA	16940-66-2 NaBH ₄
	PRO	A	296768-82-6
	SOL	64-17-5	EtOH
	NTE	0°	

L8 ANSWER 1 OF 23 HCAPLUS COPYRIGHT 2009 ACS on STN
 ACCESSION NUMBER: 2005:1196549 HCAPLUS Full-text
 DOCUMENT NUMBER: 143:440617
 TITLE: Preparation of 5-pregnanones as intermediates for squalamine
 INVENTOR(S): Koyakumaru, Kenichi
 PATENT ASSIGNEE(S): Kuraray Co., Ltd., Japan
 SOURCE: Jpn. Kokai Tokkyo Koho, 16 pp.
 CODEN: JKXXAF
 DOCUMENT TYPE: Patent
 LANGUAGE: Japanese
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2005314405	A	20051110	JP 2005-100945	20050331 <--
PRIORITY APPLN. INFO.:			JP 2004-108460	A 20040331 <--
OTHER SOURCE(S):		MARPAT 143:440617		
ED Entered STN: 10 Nov 2005				
GI				



AB 5-Pregnanones I (R11, R12 = H, protecting group) are prepared by selective hydrogenation of C-C double bond of their corresponding 5-pregn-1-en-3-ones. Thus, (20S)-7 α ,21-dihydroxy-20-methyl-5 α -pregn-1-en-3-one was hydrogenated over Pd/C at 50° for 22 h in THF to give 95% (20S)-7 α ,21-dihydroxy-20-methyl-5 α -pregnan-3-one.

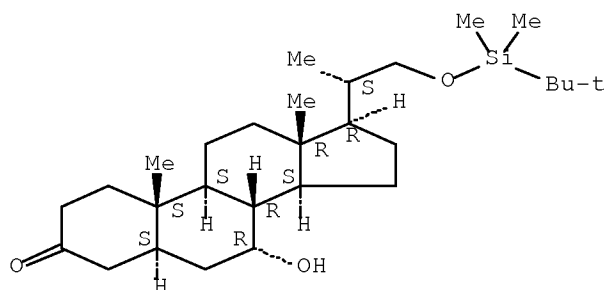
IT 303178-20-3F

RL: IMF (Industrial manufacture); RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
 (preparation of pregnanones as intermediates for squalamine from pregnenones)

RN 303178-20-3 HCAPLUS

CN Pregnan-3-one, 21-[[[(1,1-dimethylethyl)dimethylsilyl]oxy]-7-hydroxy-20-methyl-, (5 α ,7 α ,20S)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



IT 301695-48-7P

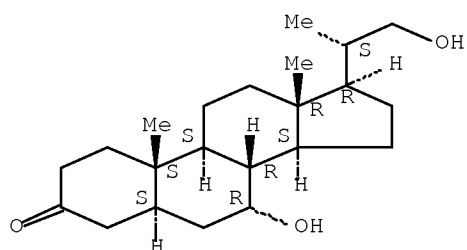
RL: IMF (Industrial manufacture); SPN (Synthetic preparation); PREP (Preparation)

(preparation of pregnanones as intermediates for squalamine from pregnenones)

RN 301695-48-7 HCAPLUS

CN Pregnan-3-one, 7,21-dihydroxy-20-methyl-, (5 α ,7 α ,20S)- (9CI)
(CA INDEX NAME)

Absolute stereochemistry.



IT 866488-96-2P 866562-46-1P

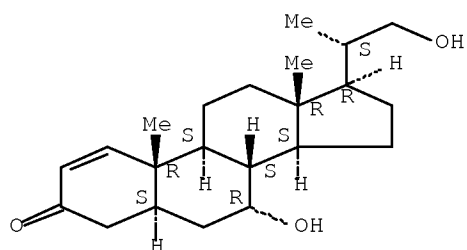
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(preparation of pregnanones as intermediates for squalamine from pregnenones)

RN 866488-96-2 HCAPLUS

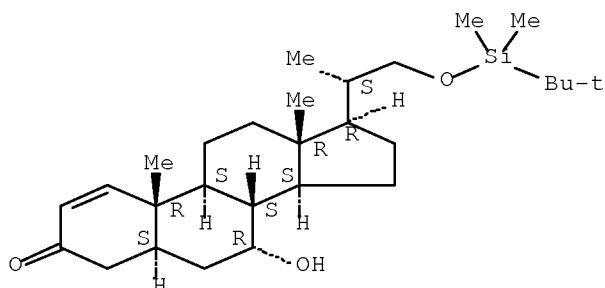
CN Pregn-1-en-3-one, 7,21-dihydroxy-20-methyl-, (5 α ,7 α ,20S)-
(9CI) (CA INDEX NAME)

Absolute stereochemistry.



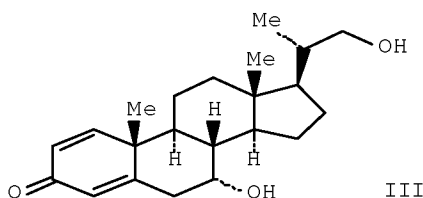
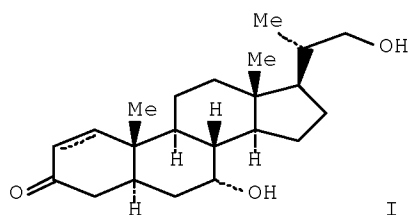
RN 866562-46-1 HCAPLUS
 CN Pregn-1-en-3-one, 21-[[(1,1-dimethylethyl)dimethylsilyl]oxy]-7-hydroxy-20-methyl-, (5 α ,7 α ,20S)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



L8 ANSWER 2 OF 23 HCAPLUS COPYRIGHT 2009 ACS on STN
 ACCESSION NUMBER: 2005:1125857 HCAPLUS [Full-text](#)
 DOCUMENT NUMBER: 143:387238
 TITLE: Preparation of 5 α -pregnane derivative as intermediate for squalamine
 INVENTOR(S): Nakasawa, Makoto; Koyakumaru, Kenichi
 PATENT ASSIGNEE(S): Kuraray Co., Ltd., Japan
 SOURCE: Jpn. Kokai Tokkyo Koho, 11 pp.
 CODEN: JKXXAF
 DOCUMENT TYPE: Patent
 LANGUAGE: Japanese
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2005289901	A	20051020	JP 2004-108416	20040331 <--
PRIORITY APPLN. INFO.:			JP 2004-108416	20040331 <--
ED Entered STN: 20 Oct 2005				
GI				



AB Title derivative I (the broken line is none: II) is prepared by treatment of pregnadienone derivative III with alkali metals or alkaline earth metals in the presence of amines and/or ammonia, followed by reduction of resulting mixture of I (the broken line is bond: IV) and II. Thus, hydrogenation of III with Li and ammonia at -40° in THF gave II-IV mixture, which was hydrogenated over Pd/C to give II.

IT ~~866488-96-2P~~

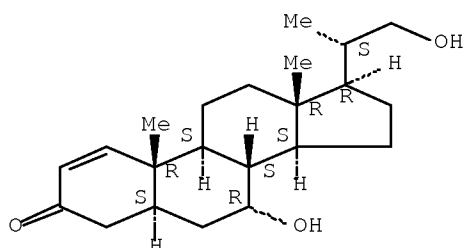
RL: IMF (Industrial manufacture); RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(preparation of 5α -pregnane derivative as intermediate for squalamine from pregnadienone derivative)

RN 866488-96-2 HCAPLUS

CN Pregn-1-en-3-one, 7,21-dihydroxy-20-methyl-, ($5\alpha, 7\alpha, 20S$)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



IT 301695-48-7P

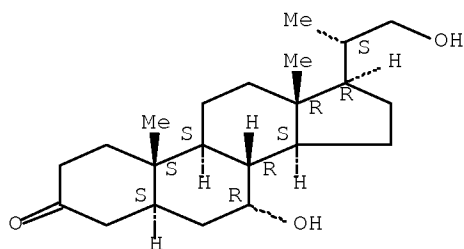
RL: IMF (Industrial manufacture); SPN (Synthetic preparation); PREP (Preparation)

(preparation of 5α -pregnane derivative as intermediate for squalamine from pregnadienone derivative)

RN 301695-48-7 HCAPLUS

CN Pregnan-3-one, 7,21-dihydroxy-20-methyl-, ($5\alpha, 7\alpha, 20S$)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



L8 ANSWER 3 OF 23 HCAPLUS COPYRIGHT 2009 ACS on STN
 ACCESSION NUMBER: 2005:1106865 HCAPLUS Full-text
 DOCUMENT NUMBER: 143:387237

TITLE: Process for the preparation of 5α -pregnane derivative via reduction of carbon-carbon double bond

Serial No.:10/594,163

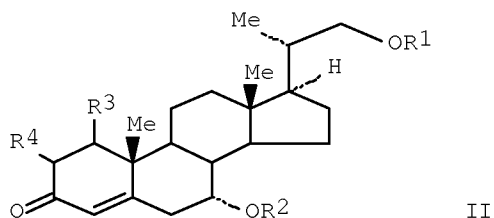
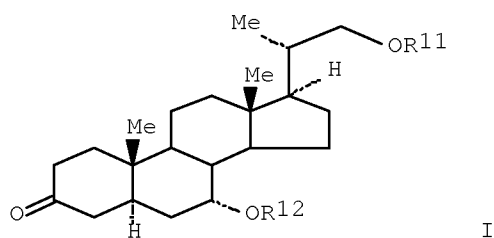
in pregn-4-en-3-one compound
 INVENTOR(S): Sugioka, Takashi; Ohzono, Shigeo; Koyakumaru, Kenichi; Nakagawa, Naoshi
 PATENT ASSIGNEE(S): Kuraray Co., Ltd., Japan
 SOURCE: PCT Int. Appl., 27 pp.
 CODEN: PIXXD2
 DOCUMENT TYPE: Patent
 LANGUAGE: Japanese
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2005095434	A1	20051013	WO 2005-JP6828	20050331 <--
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SM, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW RW: BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
EP 1743902	A1	20070117	EP 2005-728917	20050331 <--
R: AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LI, LT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR				
CN 1934124	A	20070321	CN 2005-80009530	20050331 <--
US 20070149494	A1	20070628	US 2006-594164	20060926 <--
IN 2006CN03994	A	20070706	IN 2006-CN3994	20061031 <--
PRIORITY APPLN. INFO.:			JP 2004-108419	A 20040331 <--
			WO 2005-JP6828	W 20050331 <--

OTHER SOURCE(S): MARPAT 143:387237

ED Entered STN: 14 Oct 2005

GI



AB A process for the preparation of compound I [R11, R12 = H, protecting group of hydroxy], characterized by reduction a pregnane derivative II [R1 = protecting group of hydroxy; R2 = H, protecting group of hydroxy; R3 and R4 represent hydrogen atoms or combine together to form a bond.] with alkali metals or alkaline earth metals in the presence of a proton donor, amine and/or ammonia, was disclosed. For example, to a solution of (20S)-21-tert-butyltrimethylsilyloxy-7 α -hydroxy-20-methylpregn-4-en-3-one (5.00 g) and tert-butanol (1.78 g) in THF (100 mL) was added liquid ammonia (100 mL) at -50 °C. Then, Li metal (0.17 g) was added, while maintaining the reaction temperature between -50 and -40 °C, the reaction was stirred -40 °C for 3 h. The resulting reaction was treated with ammonium sulfate (1.59 g) followed by removal of ammonia, aqueous work-up and silica-gel purification to give (20S)-21-tert-butyltrimethylsilyloxy-7 α -hydroxy-20-methyl-5 α -pregn-3-one in 96% yield. Of note compds. I are useful synthetic intermediates for the preparation of squalamine.

IT 301695-48-7P

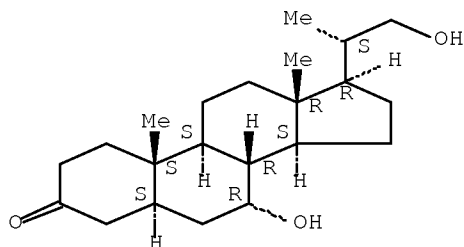
RL: SPN (Synthetic preparation); PREP (Preparation)

(preparation of (20S)-7 α ,21-dihydroxy-20-methyl-5 α -pregn-3-one via reduction and desilylation)

RN 301695-48-7 HCAPLUS

CN Pregnan-3-one, 7,21-dihydroxy-20-methyl-, (5 α ,7 α ,20S)- (9CI)
(CA INDEX NAME)

Absolute stereochemistry.



IT 303178-20-3P

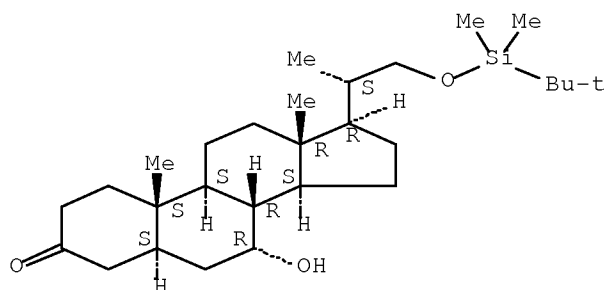
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(preparation of 5 α -pregn-3-one compound from pregn-4-en-3-one via reduction using Li metal and ammonia)

RN 303178-20-3 HCAPLUS

CN Pregnan-3-one, 21-[[[(1,1-dimethylethyl)dimethylsilyl]oxy]-7-hydroxy-20-methyl-, (5 α ,7 α ,20S)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.

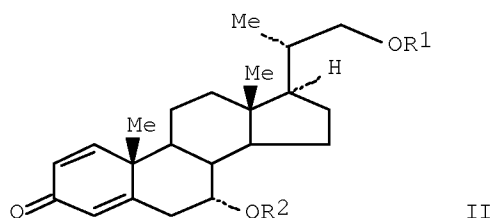
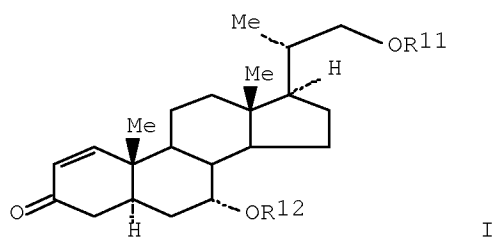


REFERENCE COUNT: 6 THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L8 ANSWER 4 OF 23 HCAPLUS COPYRIGHT 2009 ACS on STN
 ACCESSION NUMBER: 2005:1103799 HCAPLUS Full-text
 DOCUMENT NUMBER: 143:387235
 TITLE: Process for the preparation of
 5 α -pregn-1-en-3-one derivative via reduction of
 carbon-carbon double bond in pregn-1,4-dien-3-one
 compound
 INVENTOR(S): Koyakumaru, Kenichi
 PATENT ASSIGNEE(S): Kuraray Co., Ltd., Japan
 SOURCE: PCT Int. Appl., 31 pp.
 CODEN: PIXXD2
 DOCUMENT TYPE: Patent
 LANGUAGE: Japanese
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2005095433	A1	20051013	WO 2005-JP6824	20050331 <--
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SM, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW RW: BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				

PRIORITY APPLN. INFO.: JP 2004-108451 A 20040331 <--
 OTHER SOURCE(S): MARPAT 143:387235
 ED Entered STN: 14 Oct 2005
 GI



AB A process for the preparation of compds. I [R11, R12 = H, protecting group of hydroxy] from compds. II [R1 = protecting group of hydroxy; R2 = H, protecting group of hydroxy] using alkali metals or alkaline earth metals in the presence of a proton donor, amine and/or ammonia, was disclosed. For example, to a solution of (20S)-21-tert-butyldimethylsilyloxy-7 α -hydroxy-20-methylpregn-1,4-dien-3-one (10.00 g) and tert-butanol (3.23 g) in THF (170 mL) was added liquid ammonia (170 mL) at -50 °C. Then, Li metal (0.32 g) was added, while maintaining reaction temperature between -50 to -40 °C, the reaction was stirred at -40 °C for 2 h. The resulting mixture was treated with ammonium acetate (1.17 g) followed by removal of ammonia, aqueous work-up and silica-gel purification to give (20S)-21-tert-butyldimethylsilyloxy-7 α -hydroxy-20-methyl-5 α -pregn-1-en-3-one in 78% yield. Of note, compds. I are useful synthetic intermediates for the preparation of squalamine.

IT 866488-96-2P

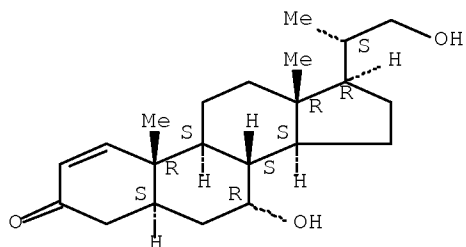
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(desilylation of (20S)-21-tert-butyldimethylsilyloxy-7 α -hydroxy-20-methylpregn-1-en-3-one)

RN 866488-96-2 HCAPLUS

CN Pregn-1-en-3-one, 7,21-dihydroxy-20-methyl-, (5 α ,7 α ,20S)-
(9CI) (CA INDEX NAME)

Absolute stereochemistry.



IT 301695-48-7P

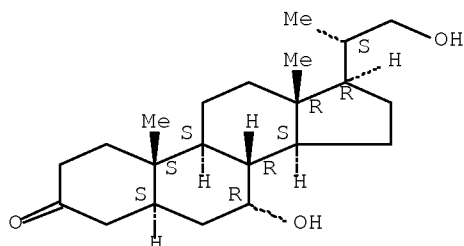
Serial No.:10/594,163

RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of (20S)-7 α ,21-dihydroxy-20-methyl-5 α -pregn-3-one
via Pd/C catalyzed hydrogenation)

RN 301695-48-7 HCAPLUS

CN Pregnan-3-one, 7,21-dihydroxy-20-methyl-, (5 α ,7 α ,20S)- (9CI)
(CA INDEX NAME)

Absolute stereochemistry.



IT 303178-20-3P

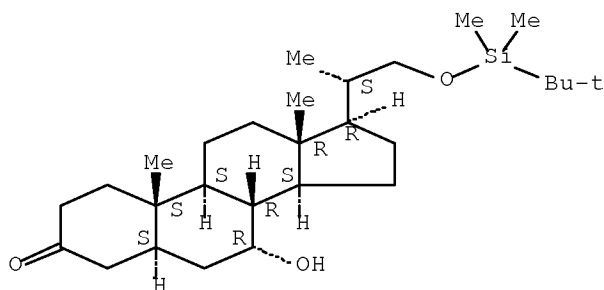
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT
(Reactant or reagent)

(preparation of 21-silyloxy-5 α -pregn-3-one compound from
21-silyloxy-5 α -pregn-1-en-3-one derivative via Pd/C catalyzed
hydrogenation)

RN 303178-20-3 HCAPLUS

CN Pregnan-3-one, 21-[[(1,1-dimethylethyl)dimethylsilyl]oxy]-7-hydroxy-20-
methyl-, (5 α ,7 α ,20S)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



IT 866562-46-1P

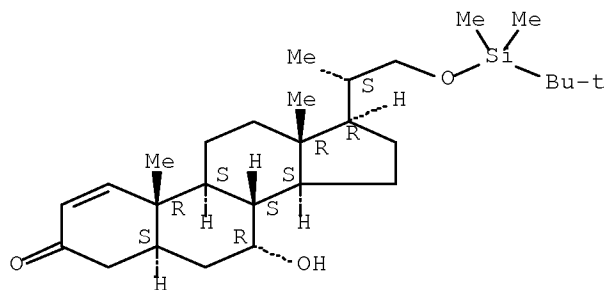
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT
(Reactant or reagent)

(preparation of pregn-1-en-3-one derivs. from pregn-1,4-dien-3-one compound
via reduction using Li metal and ammonia)

RN 866562-46-1 HCAPLUS

CN Pregn-1-en-3-one, 21-[[(1,1-dimethylethyl)dimethylsilyl]oxy]-7-hydroxy-20-
methyl-, (5 α ,7 α ,20S)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



REFERENCE COUNT: 6 THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L8 ANSWER 5 OF 23 HCAPLUS COPYRIGHT 2009 ACS on STN
 ACCESSION NUMBER: 2005:1103798 HCAPLUS Full-text
 DOCUMENT NUMBER: 143:387234

TITLE: Process for the preparation of 5 α -pregnane derivative via reduction of carbon-carbon double bond in 5 α -pregn-1-en-3-one

INVENTOR(S): Koyakumaru, Kenichi
 PATENT ASSIGNEE(S): Kuraray Co., Ltd., Japan
 SOURCE: PCT Int. Appl., 32 pp.
 CODEN: PIXXD2

DOCUMENT TYPE: Patent
 LANGUAGE: Japanese
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2005095432	A1	20051013	WO 2005-JP6819	20050331 <--
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SM, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW RW: BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
EP 1731526	A1	20061213	EP 2005-728912	20050331 <--
R: AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LI, LT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR				
CN 1938331	A	20070328	CN 2005-80010329	20050331 <--
US 20070203106	A1	20070830	US 2006-594401	20060926 <--
IN 2006CN04001	A	20070629	IN 2006-CN4001	20061031 <--
PRIORITY APPLN. INFO.:			JP 2004-108443	A 20040331 <--
			WO 2005-JP6819	W 20050331 <--

OTHER SOURCE(S): MARPAT 143:387234
 ED Entered STN: 14 Oct 2005
 GI

* STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY - AVAILABLE VIA OFFLINE PRINT *

AB Process for the preparation of compound I [R11, R12 = H, protecting group of hydroxy], characterized by selectively reducing a carbon-carbon double bond in a mixture of 5 α -pregnane derivs. II [R1, R2 = H, protecting group of hydroxy] and III [R1, R2 = same as above], was disclosed. For example, a mixture of (20S)-7 α ,21-dihydroxy-20-methyl-5 α -pregn-3-one (2.76 g) and (20S)-7 α ,21-dihydroxy-20-methyl-5 α -pregn-1-en-3-one (0.46 g) in THF was hydrogenated in the presence of 10% Pd/C (50 mg) under H₂ atmosphere at 50 °C for 5 h to give (20S)-7 α ,21-dihydroxy-20-methyl-5 α -pregn-3-one (3.06 g). Of note, compds. I are useful synthetic intermediates for the preparation of squalamine.

IT ~~866488-96-2P~~

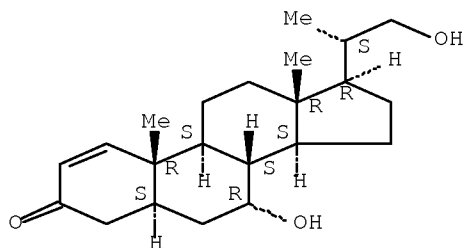
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(desilylation of a mixture of (20S)-21-tert-butyldimethylsilyloxy-7 α -hydroxy-20-methyl-5 α -pregn-1-en-3-one and (20S)-21-tert-Butyldimethylsilyloxy-7 α -hydroxy-20-methyl-5 α -pregn-3-one)

RN 866488-96-2 HCAPLUS

CN Pregn-1-en-3-one, 7,21-dihydroxy-20-methyl-, (5 α ,7 α ,20S)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



IT ~~301695-48-7P~~

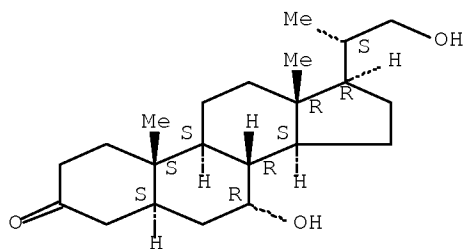
RL: SPN (Synthetic preparation); PREP (Preparation)

(preparation of (20S)-7 α ,21-dihydroxy-20-methyl-5 α -pregn-3-one via desilylation of silylated pregn-one derivs. or hydrogenation of pregnenone derivs.)

RN 301695-48-7 HCAPLUS

CN Pregnan-3-one, 7,21-dihydroxy-20-methyl-, (5 α ,7 α ,20S)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



IT 303178-20-3P

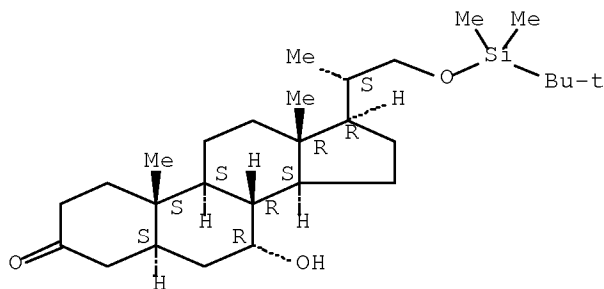
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(reduction of (20S)-21-tert-butyldimethylsilyloxy-7 α -hydroxy-20-methylpregn-1,4-dien-3-one using Li metal and ammonia)

RN 303178-20-3 HCAPLUS

CN Pregn-3-one, 21-[[[(1,1-dimethylethyl)dimethylsilyl]oxy]-7-hydroxy-20-methyl-, (5 α ,7 α ,20S)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



IT 866562-46-1P

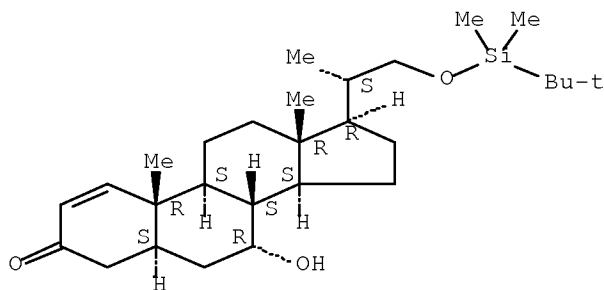
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(reduction of (20S)-7 α ,21-dihydroxy-20-methylpregn-1,4-dien-3-one using Li metal and ammonia)

RN 866562-46-1 HCAPLUS

CN Pregn-1-en-3-one, 21-[[[(1,1-dimethylethyl)dimethylsilyl]oxy]-7-hydroxy-20-methyl-, (5 α ,7 α ,20S)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



REFERENCE COUNT: 10 THERE ARE 10 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L8 ANSWER 6 OF 23 HCAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 2005:1103797 HCAPLUS Full-text

DOCUMENT NUMBER: 143:387233

TITLE: Method for producing 5 α -pregnane derivative

INVENTOR(S): Koyakumaru, Kenichi; Sugioka, Takashi; Ohzono, Shigeo; Nakagawa, Naoshi

PATENT ASSIGNEE(S): Kuraray Co., Ltd., Japan

SOURCE: PCT Int. Appl., 33 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent

LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

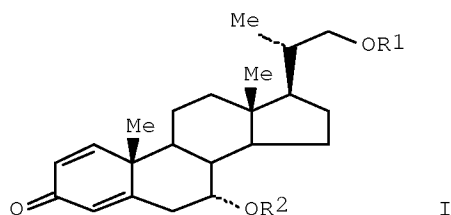
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2005095431	A1	20051013	WO 2005-JP6818	20050331 <--
W:	AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SM, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW			
RW:	BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG			
CN 1938330	A	20070328	CN 2005-80010110	20050331 <--
EP 1767540	A1	20070328	EP 2005-728889	20050331 <--
R:	AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LI, LT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR			
US 20070197490	A1	20070823	US 2006-594163	20060926 <--
IN 2006CN03996	A	20070706	IN 2006-CN3996	20061031 <--
PRIORITY APPLN. INFO.:			JP 2004-108434	A 20040331 <--
			WO 2005-JP6818	W 20050331 <--

OTHER SOURCE(S): MARPAT 143:387233

ED Entered STN: 14 Oct 2005

GI



AB 5 α -Pregna-3-one derivs. and 5 α -pregna-1-en-3-one derivs. are prepared by reacting a pregnane derivative represented by the general formula I [R1 = OH-protecting group; R2 = H, OH-protecting group] with a metal selected from alkali metals and alkaline earth metals in the presence of a proton donor, an amine and/or ammonia. The title compds. are intermediates for squalamine. Thus, (20S)-21-tert-butyltrimethylsilyloxy-7 α -hydroxy-20-methyl-5 α -pregna-3-one and (20S)-21-tert-butyltrimethylsilyloxy-7 α -hydroxy-20-methyl-5 α -pregna-1-en-3-one were prepared by the title method.

IT 303178-20-3F 866562-46-1F

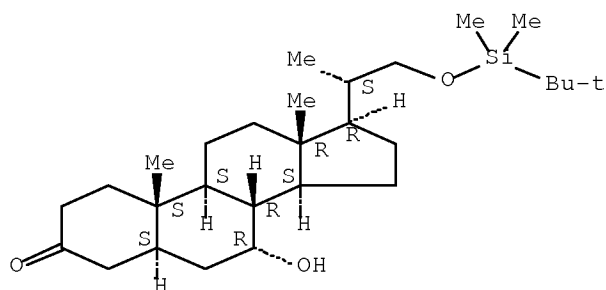
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(method for producing 5 α -pregnane derivs. by reduction of methylpregna-1,4-dien-3-one derivs.)

RN 303178-20-3 HCAPLUS

CN Pregnan-3-one, 21-[[[(1,1-dimethylethyl)dimethylsilyl]oxy]-7-hydroxy-20-methyl-, (5 α ,7 α ,20S)- (9CI) (CA INDEX NAME)

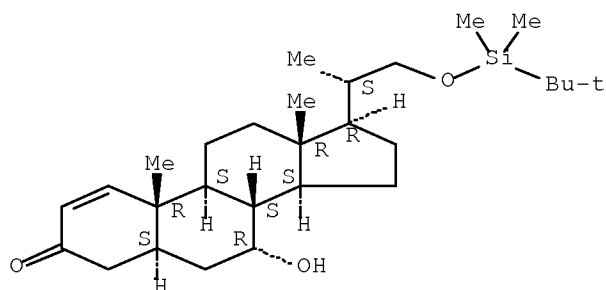
Absolute stereochemistry.



RN 866562-46-1 HCAPLUS

CN Pregn-1-en-3-one, 21-[[[(1,1-dimethylethyl)dimethylsilyl]oxy]-7-hydroxy-20-methyl-, (5 α ,7 α ,20S)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



IT 301695-48-7P 866488-96-2P

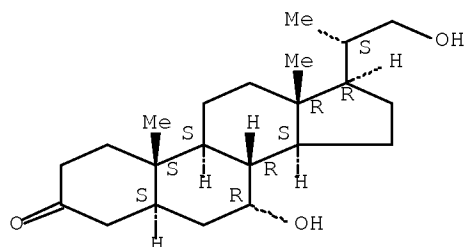
RL: SPN (Synthetic preparation); PREP (Preparation)

(method for producing 5 α -pregnane derivs. by reduction of methylpregna-1,4-dien-3-one derivs.)

RN 301695-48-7 HCAPLUS

CN Pregnan-3-one, 7,21-dihydroxy-20-methyl-, (5 α ,7 α ,20S)- (9CI)
(CA INDEX NAME)

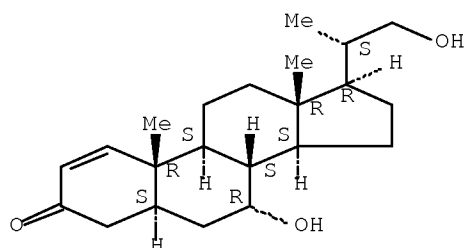
Absolute stereochemistry.



RN 866488-96-2 HCAPLUS

CN Pregn-1-en-3-one, 7,21-dihydroxy-20-methyl-, (5 α ,7 α ,20S)-
(9CI) (CA INDEX NAME)

Absolute stereochemistry.



REFERENCE COUNT:

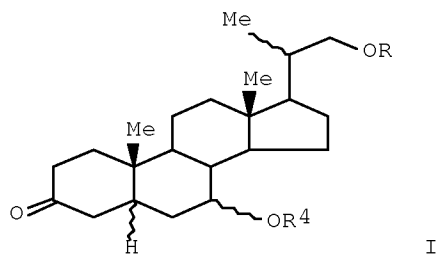
6

THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS
RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

Serial No.:10/594,163

ACCESSION NUMBER: 2003:491250 HCAPLUS Full-text
DOCUMENT NUMBER: 139:69426
TITLE: Process for producing pregnane derivative
INVENTOR(S): Nakazawa, Makoto; Ohzono, Shigeo
PATENT ASSIGNEE(S): Kuraray Co., Ltd., Japan
SOURCE: PCT Int. Appl., 35 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2003051904	A1	20030626	WO 2002-JP11547	20021106 <--
W: CA, CN, HU, IN, MX, US				
RW: AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, SK, TR				
JP 2003246790	A	20030902	JP 2002-322581	20021106 <--
PRIORITY APPLN. INFO.:			JP 2001-386808	A 20011219 <--
OTHER SOURCE(S):			MARPAT 139:69426	
ED Entered STN: 27 Jun 2003				
GI				



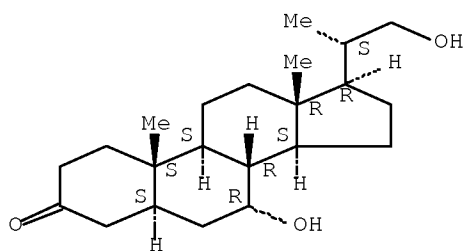
AB This document discloses a process for producing a 21-hydroxypregnane derivative represented by the general formula I [R = H; R4 represents a hydroxy-protecting group.] characterized by protecting the 7-position hydroxy group of a compound represented by the formula I [R = SiR1R2R3; R1, R2, and R3 each independently represents optionally substituted alkyl, alkenyl, alkynyl, aryl, or aralkyl; and R4 = H] and subsequently eliminating the 21-position protective silyl group. An intermediate for squalamine can be efficiently produced by the title process.

IT ~~301695-48-7P 303178-20-3P 550372-80-0P~~
RL: IMF (Industrial manufacture); RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(process for producing pregnane derivative)

RN 301695-48-7 HCAPLUS

CN Pregnan-3-one, 7,21-dihydroxy-20-methyl-, (5 α ,7 α ,20S)- (9CI)
(CA INDEX NAME)

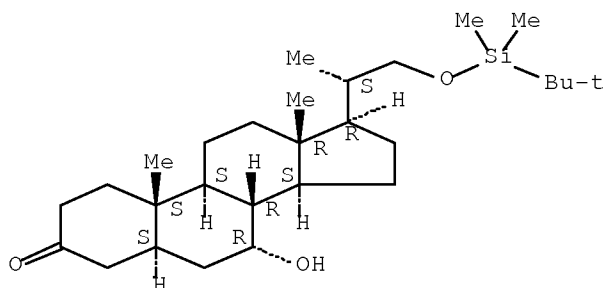
Absolute stereochemistry.



RN 303178-20-3 HCAPLUS

CN Pregnan-3-one, 21-[[[(1,1-dimethylethyl)dimethylsilyl]oxy]-7-hydroxy-20-methyl-, (5 α ,7 α ,20S)- (9CI) (CA INDEX NAME)

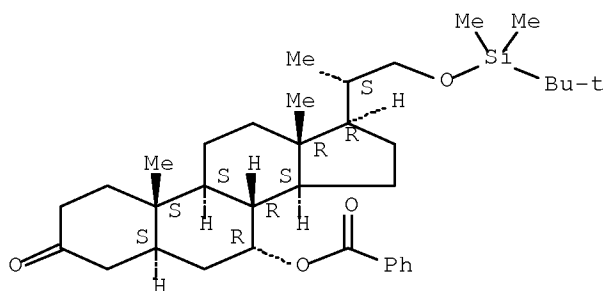
Absolute stereochemistry.



RN 550372-80-0 HCAPLUS

CN Pregnan-3-one, 7-(benzoyloxy)-21-[[[(1,1-dimethylethyl)dimethylsilyl]oxy]-20-methyl-, (5 α ,7 α ,21S)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



IT 403854-17-1P

RL: IMF (Industrial manufacture); SPN (Synthetic preparation); PREP (Preparation)

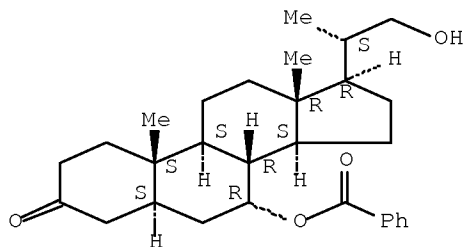
(process for producing pregnane derivative)

RN 403854-17-1 HCAPLUS

CN Pregnan-3-one, 7-(benzoyloxy)-21-hydroxy-20-methyl-,

(5 α , 7 α , 20S)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



REFERENCE COUNT: 3 THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L8 ANSWER 8 OF 23 HCAPLUS COPYRIGHT 2009 ACS on STN
 ACCESSION NUMBER: 2002:185147 HCAPLUS Full-text
 DOCUMENT NUMBER: 136:247742
 TITLE: Process for the preparation of pregnane derivatives
 INVENTOR(S): Nakazawa, Makoto
 PATENT ASSIGNEE(S): Kuraray Co., Ltd., Japan
 SOURCE: PCT Int. Appl., 52 pp.
 CODEN: PIXXD2
 DOCUMENT TYPE: Patent
 LANGUAGE: Japanese
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2002020552	A1	20020314	WO 2001-JP7639	20010904 <--
W: CA, CN, HU, IN, MX, US				
RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR				
JP 2002201199	A	20020716	JP 2001-261586	20010830 <--
CA 2416850	A1	20030120	CA 2001-2416850	20010904 <--
EP 1325928	A1	20030709	EP 2001-961343	20010904 <--
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, FI, CY, TR				
US 20030181742	A1	20030925	US 2003-363405	20030304 <--
PRIORITY APPLN. INFO.:			JP 2000-273387	A 20000908 <--
			WO 2001-JP7639	W 20010904 <--
OTHER SOURCE(S): CASREACT 136:247742; MARPAT 136:247742				
ED Entered STN: 15 Mar 2002				
GI				

* STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY - AVAILABLE VIA OFFLINE PRINT *

AB A process for the preparation of pregnane derivs. [I; R1 = H; R2 = protecting group; R3, R4 independently = alkyl, aryl, alkenyl, alkynyl; R3R4 = alkylene] which comprises reacting a compound II with an alkali metal or an alkaline earth metal in the presence of ammonia or an amine to obtain a compound III

(R1 = H; R2 = H), protecting the hydroxyl groups of the compound III to obtain a compound III (R1 = protecting group; R2 = protecting group) protecting the compound III (R1 = protecting group; R2 = protecting group) at the 3-position to obtain a compound I (R1 = protecting group; R2 = protecting group; R3, R4 independently = alkyl, aryl, alkenyl, alkynyl; R3R4 = alkylene), and subjecting the compound I (R1 = protecting group; R2 = protecting group; R3, R4 independently = alkyl, aryl, alkenyl, alkynyl; R3R4 = alkylene) to solvolysis to obtain a compound I (R1 = H; R2 = protecting group; R3, R4 independently = alkyl, aryl, alkenyl, alkynyl; R3R4 = alkylene) and compound III (R1 = H; R2 = H). Thus, the title compound I (R1 = H; R2 = C₆H₅CO; R3R4 = CH₂CH₂) was prepared effectively from (20S)-7- α -hydroxy-3-oxo-pregna-1,4-diene-20-carboxaldehyde and C₆H₅COCl via hydrogenation and ethylene glycol and C₆H₅COCl O-protection and was useful as intermediate for squalamine preparation

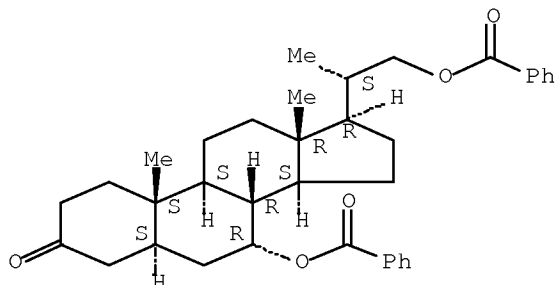
IT ~~403854-15-9P~~ 403854-17-1P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(process for the preparation of pregnane derivs.)

RN 403854-15-9 HCAPLUS

CN Pregnan-3-one, 7,21-bis(benzoyloxy)-20-methyl-, (5 α ,7 α ,20S)-
(9CI) (CA INDEX NAME)

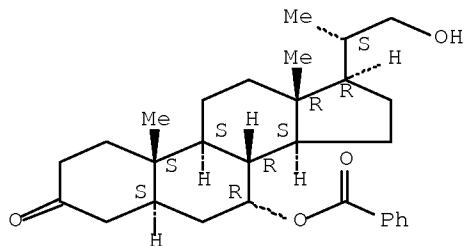
Absolute stereochemistry.



RN 403854-17-1 HCAPLUS

CN Pregnan-3-one, 7-(benzoyloxy)-21-hydroxy-20-methyl-,
(5 α ,7 α ,20S)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



IT 301695-48-7P

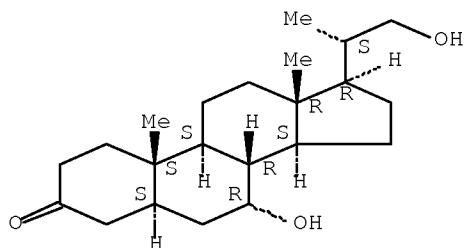
RL: SPN (Synthetic preparation); PREP (Preparation)

(process for the preparation of pregnane derivs.)

RN 301695-48-7 HCAPLUS

CN Pregnan-3-one, 7,21-dihydroxy-20-methyl-, (5 α ,7 α ,20S)- (9CI)
(CA INDEX NAME)

Absolute stereochemistry.



REFERENCE COUNT: 12 THERE ARE 12 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L8 ANSWER 9 OF 23 HCAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 2001:872204 HCAPLUS Full-text

DOCUMENT NUMBER: 136:167554

TITLE: Synthesis and antimicrobial activity of new
3 α -Hydroxy-23,24-bisnorcholane polyamine
carbamates

AUTHOR(S): Kim, Hong-Seok; Kwon, Kyung-Chan; Kim, Ki Soo; Lee,
Cheol Hae

CORPORATE SOURCE: Department of Industrial Chemistry, Kyungpook National
University, Taegu, 702-701, S. Korea

SOURCE: Bioorganic & Medicinal Chemistry Letters (2001
, 11(23), 3065-3068

CODEN: BMCLE8; ISSN: 0960-894X

PUBLISHER: Elsevier Science Ltd.

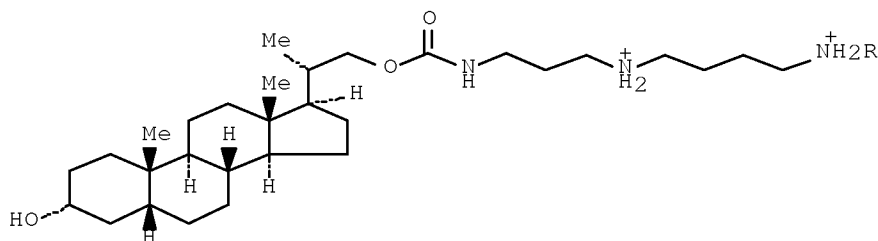
DOCUMENT TYPE: Journal

LANGUAGE: English

OTHER SOURCE(S): CASREACT 136:167554

ED Entered STN: 04 Dec 2001

GI



I

AB 3 α -Hydroxy-23,24-bisnorcholane spermidine and spermine carbamates, e.g. I.2Cl-
, have been synthesized and their antimicrobial and hemolytic activities were

evaluated. They exhibited excellent in vitro activities especially against methicillin-resistant *Staphylococcus aureus*.

IT 398138-67-5 781657-55-4

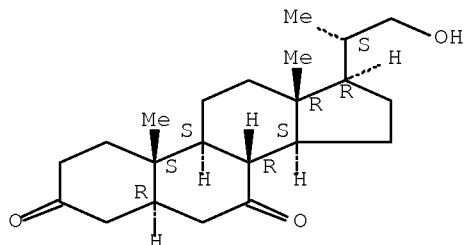
RL: RCT (Reactant); RACT (Reactant or reagent)

(preparation of bisnorcholane polyamine carbamates and antimicrobial activity against methicillin-resistant *Staphylococcus aureus*)

RN 398138-67-5 HCAPLUS

CN Pregnane-3,7-dione, 21-hydroxy-20-methyl-, (5 α ,20S)- (9CI) (CA INDEX NAME)

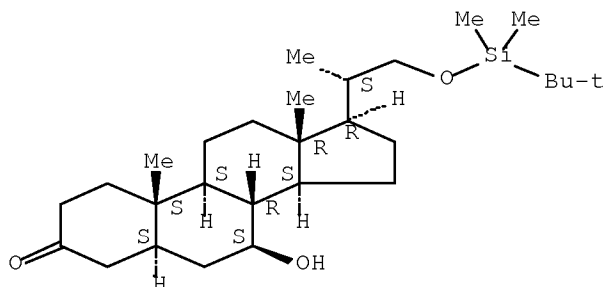
Absolute stereochemistry.



RN 781657-55-4 HCAPLUS

CN Pregnan-3-one, 21-[[[(1,1-dimethylethyl)dimethylsilyl]oxy]-7-hydroxy-20-methyl-, (5 α ,7 β ,20S)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



REFERENCE COUNT: 23 THERE ARE 23 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L8 ANSWER 10 OF 23 HCAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 2001:780935 HCAPLUS [Full-text](#)

DOCUMENT NUMBER: 135:318612

TITLE: A process for the preparation of 7 α -hydroxy 3-aminosubstituted sterols using intermediates with an unprotected 7 α -hydroxy group

INVENTOR(S): Kinney, William A.; Zhang, Xuehai; Michalak, Ronald

PATENT ASSIGNEE(S): Genaera Corporation, USA

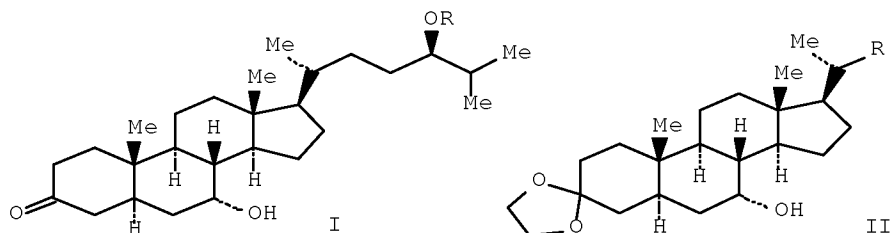
SOURCE: PCT Int. Appl., 39 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent

LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2001079255	A1	20011025	WO 2001-US12004	20010412 <--
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG				
CA 2406847	A1	20011025	CA 2001-2406847	20010412 <--
EP 1274718	A1	20030115	EP 2001-926924	20010412 <--
EP 1274718	B1	20061018		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR				
JP 2003531148	T	20031021	JP 2001-576852	20010412 <--
AT 342912	T	20061115	AT 2001-926924	20010412 <--
AU 2001253427	B2	20070208	AU 2001-253427	20010412 <--
ES 2273831	T3	20070516	ES 2001-926924	20010412 <--
US 20030171576	A1	20030911	US 2002-268660	20021011 <--
US 6933383	B2	20050823		
US 20050187202	A1	20050825	US 2005-83961	20050321 <--
PRIORITY APPLN. INFO.:			US 2000-196646P	P 20000412 <--
			WO 2001-US12004	W 20010412 <--
			US 2002-268660	A3 20021011 <--
OTHER SOURCE(S):			CASREACT 135:318612; MARPAT 135:318612	
ED Entered STN: 26 Oct 2001				
GI				



AB An efficient method for the synthesis of aminosterol compds. such as squalamine and compound 1436 is described. A method of the invention provides for regioselective oxidation and regioselective sulfonation of a fused ring system. The fused ring base can be, for example, a steroid ring base. The aminosterol compds. are effective as, among others, antibiotics, antiangiogenic agents and NHE3 inhibitors. Thus, squalamine and compound 1436 intermediate I (R = SO₃H) was prepared by the regioselective oxidation of II (R = CH₂OH) with NaOCl and TEMPO to give II (R = CHO), and regioselective sulfonation of I (R = H).

IT 301695-48-7F

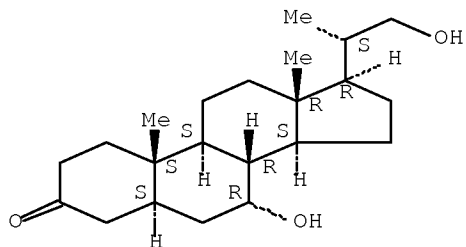
Serial No.:10/594,163

RL: IMF (Industrial manufacture); RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation of 7 α -hydroxy-3-amino-substituted steroids via regioselective oxidation and sulfonation)

RN 301695-48-7 HCAPLUS

CN Pregnan-3-one, 7,21-dihydroxy-20-methyl-, (5 α ,7 α ,20S)- (9CI)
(CA INDEX NAME)

Absolute stereochemistry.



REFERENCE COUNT: 11 THERE ARE 11 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L8 ANSWER 11 OF 23 HCAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 2000:605979 HCAPLUS Full-text

DOCUMENT NUMBER: 133:310055

TITLE: A Short Formal Synthesis of Squalamine from a Microbial Metabolite

AUTHOR(S): Kinney, William A.; Zhang, Xuehai; Williams, Jon I.; Johnston, Sean; Michalak, Ronald S.; Deshpande, Milind; Dostal, Larry; Rosazza, John P. N.

CORPORATE SOURCE: Magainin Pharmaceuticals Inc., Plymouth Meeting, PA, 19462, USA

SOURCE: Organic Letters (2000), 2(19), 2921-2922
CODEN: ORLEF7; ISSN: 1523-7060

PUBLISHER: American Chemical Society

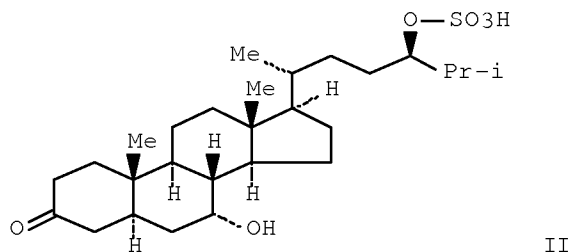
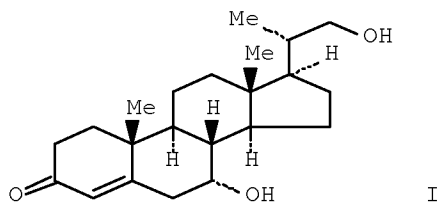
DOCUMENT TYPE: Journal

LANGUAGE: English

OTHER SOURCE(S): CASREACT 133:310055

ED Entered STN: 31 Aug 2000

GI



AB Formal synthesis of squalamine is described, utilizing the biotransformation product I, which is available in one step from com. available 3-keto-23,24-bisnorchol-4-en-22-ol. Regioselective C-22 oxidation and C-24 sulfation of the corresponding alcs. in the presence of a free C-7 alc. make for an efficient preparation of squalamine intermediate II.

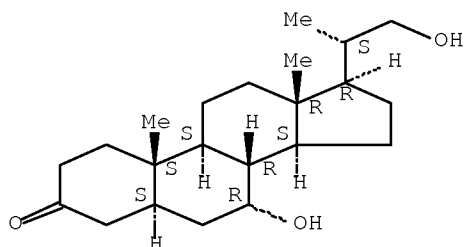
IT 301695-48-7P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(formal synthesis of squalamine from a microbial metabolite)

RN 301695-48-7 HCAPLUS

CN Pregnan-3-one, 7,21-dihydroxy-20-methyl-, (5 α ,7 α ,20S)- (9CI)
(CA INDEX NAME)

Absolute stereochemistry.



REFERENCE COUNT: 16 THERE ARE 16 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L8 ANSWER 12 OF 23 HCAPLUS COPYRIGHT 2009 ACS on STN

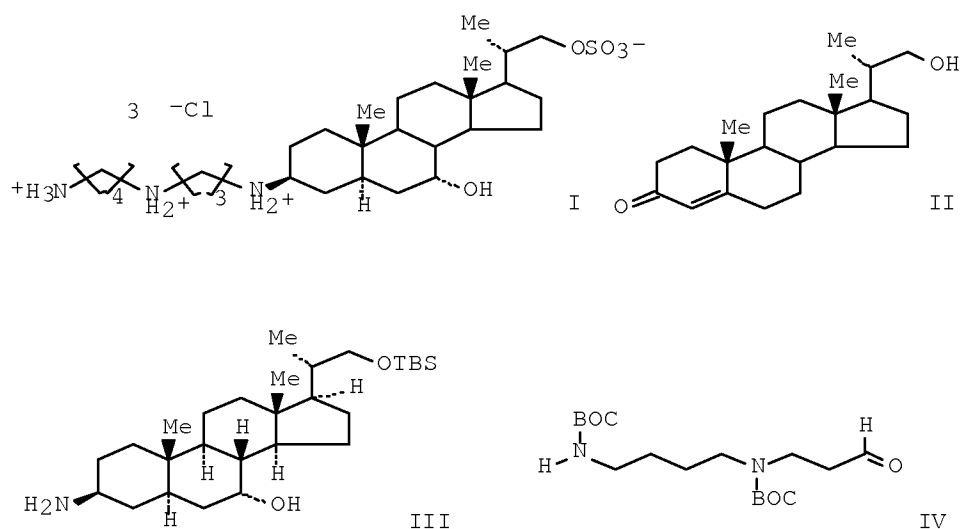
ACCESSION NUMBER: 2000:564499 HCAPLUS [Full-text](#)

DOCUMENT NUMBER: 133:335386

TITLE: Synthesis and antimicrobial activity of squalamine analogue

AUTHOR(S): Kim, H.-S.; Choi, B.-S.; Kwon, K.-C.; Lee, S.-O.;

CORPORATE SOURCE: Kwak, H. J.; Lee, C. H.
 Department of Industrial Chemistry, Kyungpook National
 University, Taegu, 702-701, S. Korea
 SOURCE: Bioorganic & Medicinal Chemistry (2000),
 8(8), 2059-2065
 CODEN: BMECEP; ISSN: 0968-0896
 PUBLISHER: Elsevier Science Ltd.
 DOCUMENT TYPE: Journal
 LANGUAGE: English
 OTHER SOURCE(S): CASREACT 133:335386
 ED Entered STN: 16 Aug 2000
 GI



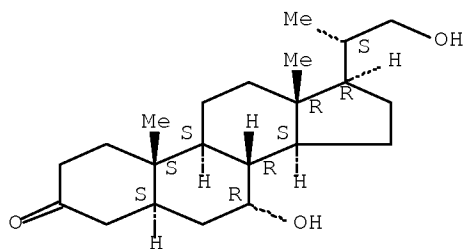
AB Synthesis and antimicrobial activity of squalamine analog (I) are reported. The synthesis of I was accomplished from bisnoralc. (II). The spermidine moiety was introduced via reductive amination of an appropriately functionalized 3β-aminosterol (III) with spermidine aldehyde (IV) utilizing sodium triacetoxyborohydride as the reducing agent. I shows weaker antimicrobial activity than squalamine.

IT 301695-48-7P 303178-20-3P
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
 (preparation and antimicrobial activity of squalamine analog)

RN 301695-48-7 HCAPLUS

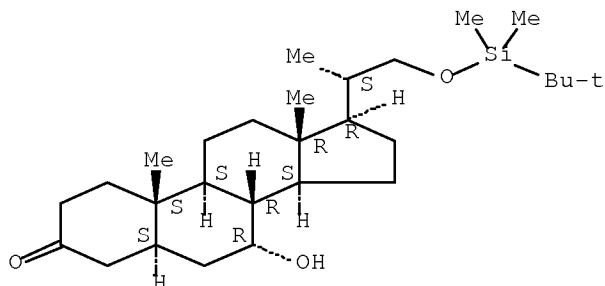
CN Pregnan-3-one, 7,21-dihydroxy-20-methyl-, (5α,7α,20S)- (9CI)
 (CA INDEX NAME)

Absolute stereochemistry.



RN 303178-20-3 HCAPLUS
 CN Pregnan-3-one, 21-[[[(1,1-dimethylethyl)dimethylsilyl]oxy]-7-hydroxy-20-methyl-, (5α,7α,20S)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



REFERENCE COUNT: 19 THERE ARE 19 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L8 ANSWER 13 OF 23 HCAPLUS COPYRIGHT 2009 ACS on STN
 ACCESSION NUMBER: 1997:540987 HCAPLUS Full-text
 DOCUMENT NUMBER: 127:229207
 ORIGINAL REFERENCE NO.: 127:44531a,44534a
 TITLE: Effect of side chain length on biotransformation, hepatic transport, and choleretic properties of chenodeoxycholy homologs in the rodent: studies with dinorchenodeoxycholic acid, norchenodeoxycholic acid, and chenodeoxycholic acid
 AUTHOR(S): Yeh, Hong-Zen; Schteingart, Claudio D.; Hagey, Lee R.; Ton-Nu, Huong-Thu; Bolder, Ulrich; Gavrilkina, Miriam A.; Steinbach, Joseph H.; Hofmann, Alan F.
 CORPORATE SOURCE: div. Gastroenterol., Dep. Med., Univ. California San Diego, San Diego, CA, USA
 SOURCE: Hepatology (Philadelphia) (1997), 26(2), 374-385
 CODEN: HPTLD9; ISSN: 0270-9139
 PUBLISHER: Saunders
 DOCUMENT TYPE: Journal
 LANGUAGE: English
 ED Entered STN: 25 Aug 1997
 AB To assess the effect of side chain length on the metabolism and physiolog. effects of homologs of chenodeoxycholic acid (CDCA), dinorCDCA, the C22 homolog, was synthesized and its hepatic biotransformation, transport

kinetics, and choleretic properties were defined in rat and hamster biliary fistula and in isolated perfused rat liver. Results were compared with those of norCDCA, the C23 homolog, and of CDCA, the natural C24 homolog. In the rat, dinorCDCA was secreted mostly in unconjugated form (the majority as dinor- α -muricholic acid); the remainder was glucuronidated. In the hamster, glucuronidation was greater, and the unconjugated fraction contained equal parts of dinor CDCA and 5 β -hydroxy-dinorCDCA. NorCDCA was glucuronidated extensively (70%, rat; 40%, hamster). CDCA, in contrast, was efficiently amidated with taurine or glycine. In the perfused liver, the initial uptake rate of all three homologs was identical; later, regurgitation and/or cholehepatic shunting of dinor-CDCA and norCDCA, but not of CDCA, occurred. In rats and hamsters with biliary fistulas, dinorCDCA and norCDCA, but not CDCA, induced a bicarbonate-rich hypercholeresis of canalicular origin. Hypercholeresis was not induced by the taurine conjugate of dinorCDCA. Hepatobiliary retention of both dinorCDCA and norCDCA occurred, consistent with efficient ductular absorption (calculated to be 94%) and cholehepatic cycling of the unmetabolized bile acids. It is concluded that dinorCDCA, as norCDCA, is inefficiently amidated, is metabolized as a xenobiotic, and induces hypercholeresis. DinorCDCA is the first dihydroxy bile acid to be identified that is secreted largely in unconjugated form in bile.

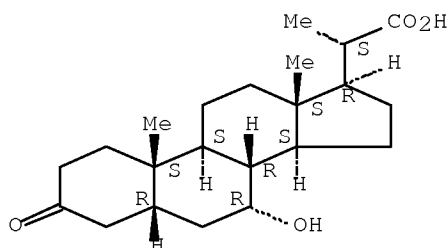
IT 195205-20-0

RL: BSU (Biological study, unclassified); MFM (Metabolic formation); BIOL (Biological study); FORM (Formation, nonpreparative)
(effect of side chain length on biotransformation and hepatic transport and choleretic properties of chenodeoxycholy homologs in rodent in relation to lipophilicity)

RN 195205-20-0 HCAPLUS

CN Pregnane-20-carboxylic acid, 7-hydroxy-3-oxo-, (5 β ,7 α)- (9CI)
(CA INDEX NAME)

Absolute stereochemistry.



REFERENCE COUNT: 54 THERE ARE 54 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L8 ANSWER 14 OF 23 HCAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 1991:602975 HCAPLUS Full-text

DOCUMENT NUMBER: 115:202975

ORIGINAL REFERENCE NO.: 115:34541a,34544a

TITLE: Bioconversion of triterpenes by mycobacteria.
Structure and conformation of the products of
degradation of 7,11-dioxodihydrolanosterol by
Mycobacterium phlei

AUTHOR(S): Jabbouri, Said; Chosson, Patricia; Tisnes, Pierre;
Rao, Renee; Servin, Philippe; Prome, Jean Claude

CORPORATE SOURCE: Cent. Rech. Biochim. Genet. Cell., CNRS, Toulouse,
31062, Fr.

SOURCE: Journal of the Chemical Society, Perkin Transactions
1: Organic and Bio-Organic Chemistry (1972-1999) (1991), (8), 1935-40
CODEN: JCPRB4; ISSN: 0300-922X

DOCUMENT TYPE: Journal
LANGUAGE: English

ED Entered STN: 15 Nov 1991

AB Although mycobacteria are unable to degrade lanosterol and dihydrolanosterol, principal components of wool fat, the transformation of some of their autoxidn. products by *M. phlei* was observed. By analogy with the mechanism of cholesterol degradation, this difference was assumed to be due to the requirement for the presence of an enone group before the side-chain can be degraded. This paper reports the spectroscopic determination of the structure of the major metabolites of 7,11-dioxodihydrolanosterol. The side-chain is degraded from 8 carbon atoms to 3, the terminal carbon atom being oxidized to a primary alc. or a Me ester. The tetracyclic skeleton can undergo regioselective oxidation-reduction modifications at the 3- and 7-position. Their conformational anal., carried out by 2D-NMR methods, indicates a chair form for ring A of 3 β -hydroxy derivs., while it is highly deformed for 3-keto compds. as predicted formerly by D. A. Dougherty, et al. (1979) for this lanostane series.

IT 136842-50-7 136842-51-8 136842-53-0
136842-54-1

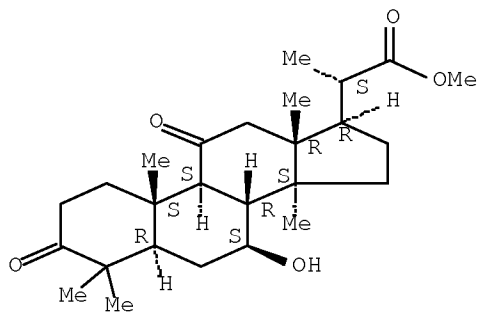
RL: BIOL (Biological study)

(formation and conformation and structure of, of *Mycobacterium phlei*, as dioxodihydrolanosterol degradation product)

RN 136842-50-7 HCAPLUS

CN Pregnane-20-carboxylic acid, 7-hydroxy-4,4,14-trimethyl-3,11-dioxo-, methyl ester, (5 α ,7 β ,20S)- (9CI) (CA INDEX NAME)

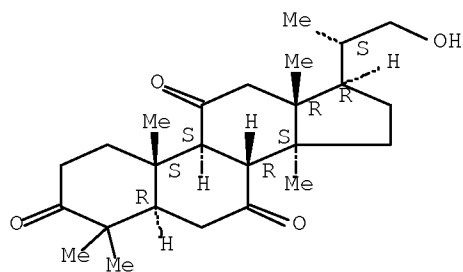
Absolute stereochemistry.



RN 136842-51-8 HCAPLUS

CN Pregnane-3,7,11-trione, 21-hydroxy-4,4,14,20-tetramethyl-, (5 α ,20S)- (9CI) (CA INDEX NAME)

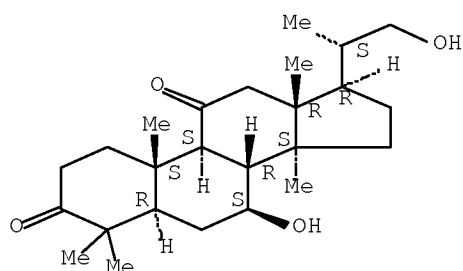
Absolute stereochemistry.



RN 136842-53-0 HCAPLUS

CN Pregnane-3,11-dione, 7,21-dihydroxy-4,4,14,20-tetramethyl-,
(5 α ,7 β ,20S)- (9CI) (CA INDEX NAME)

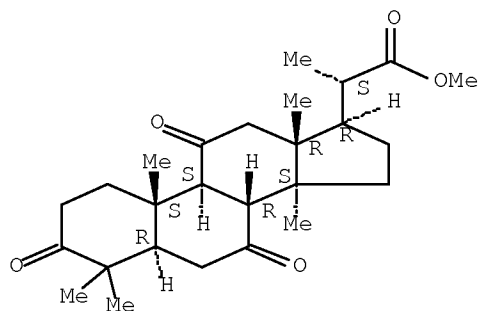
Absolute stereochemistry.



RN 136842-54-1 HCAPLUS

CN Pregnane-20-carboxylic acid, 4,4,14-trimethyl-3,7,11-trioxo-, methyl
ester, (5 α ,20S)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



L8 ANSWER 15 OF 23 HCAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 1981:407603 HCAPLUS [Full-text](#)

DOCUMENT NUMBER: 95:7603

ORIGINAL REFERENCE NO.: 95:1447a,1450a

TITLE: Chenodeoxycholic acid and intermediate products

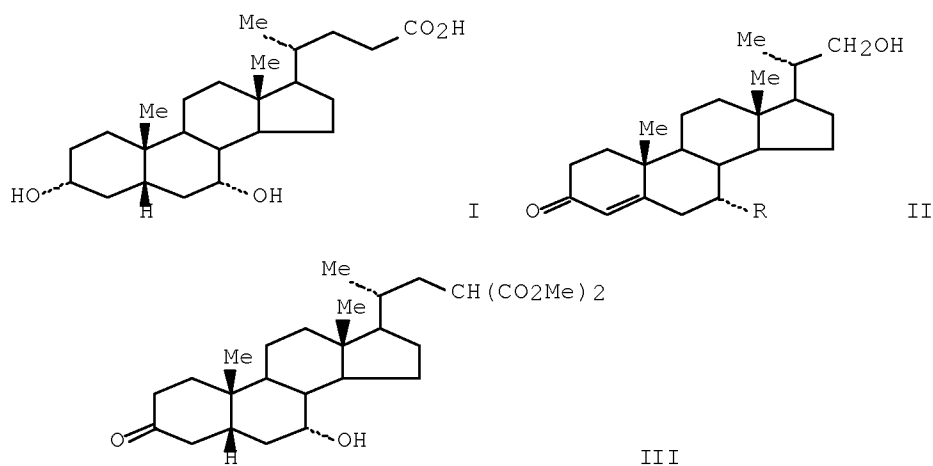
Serial No.:10/594,163

INVENTOR(S): Despreaux, Carl; Narwid, Thomas Albert; Palleroni, Norberto J.; Uskokovic, Milan R.
 PATENT ASSIGNEE(S): Hoffmann-La Roche, F., und Co. A.-G., Switz.
 SOURCE: Eur. Pat. Appl., 37 pp.
 CODEN: EPXXDW
 DOCUMENT TYPE: Patent
 LANGUAGE: German
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 18515	A2	19801112	EP 1980-101893	19800409 <--
EP 18515	A3	19810107		
EP 18515	B1	19821027		
R: AT, BE, CH, DE, FR, GB, IT, NL				
US 4230625	A	19801028	US 1979-29420	19790412 <--
US 4301246	A	19811117	US 1980-113019	19800118 <--
AT 1710	T	19821115	AT 1980-101893	19800409 <--
JP 56008399	A	19810128	JP 1980-47936	19800411 <--
PRIORITY APPLN. INFO.:			US 1979-29420	A 19790412 <--
			EP 1980-101893	A 19800409 <--

OTHER SOURCE(S): CASREACT 95:7603; MARPAT 95:7603

ED Entered STN: 12 May 1984
 GI



AB Chenodeoxycholic acid (I) was prepared from oxodinorcholenoic acid II (R = H). Thus, fermentation of II (R = H) with Botryodiplodia theobromae gave II (R = OH), which underwent consecutive hydrogenation, tosylation, and substitution reaction with CH₂(CO₂Me)₂ to give norcholanededicarboxylate III. NaBH₄ reduction of III and subsequent saponification-decarboxylation gave I.

IT 77530-53-1P

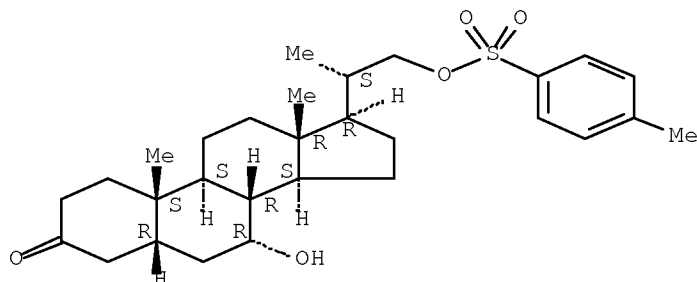
RL: SPN (Synthetic preparation); PREP (Preparation)
 (preparation and substitution reaction with di-Me malonate)

RN 77530-53-1 HCAPLUS

CN Pregnan-3-one, 7-hydroxy-20-methyl-21-[[(4-methylphenyl) sulfonyl]oxy]-,

(5 β , 7 α , 20S)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



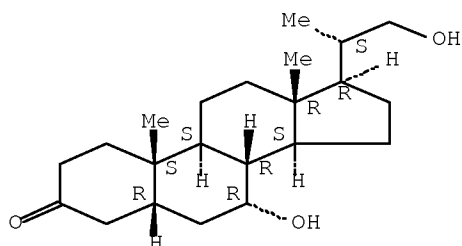
IT 77530-52-0P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent) (preparation and tosylation of)

RN 77530-52-0 HCAPLUS

CN Pregnan-3-one, 7,21-dihydroxy-20-methyl-, (5 β , 7 α , 20S)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



L8 ANSWER 16 OF 23 HCAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 1964:61153 HCAPLUS [Full-text](#)

DOCUMENT NUMBER: 60:61153

ORIGINAL REFERENCE NO.: 60:10750g-h

TITLE: Helvolic acid

AUTHOR(S): Okuda, Shigenobu; Iwasaki, Shigeo; Tsuda, Kyosuke; Sano, Yoshimoto; Hata, Toju; Udagawa, Shunichi; Nakayama, Yuya; Yamaguchi, Hiroshi

CORPORATE SOURCE: Univ. Tokyo

SOURCE: Chemical & Pharmaceutical Bulletin (1964), 12(1), 121-4

CODEN: CPBTAL; ISSN: 0009-2363

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

ED Entered STN: 22 Apr 2001

GI For diagram(s), see printed CA Issue.

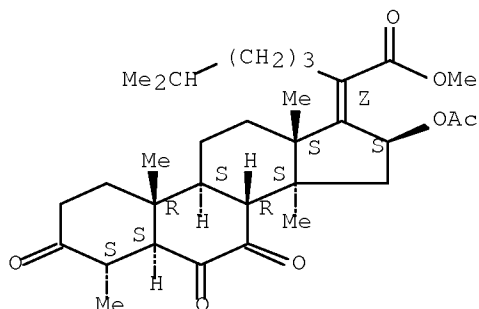
AB I was proposed as a partial structure for helvolic acid on the basis of chemical and spectral evidence. It was suggested that the remaining Me group was probably at C-14.

IT 107277-22-5, 29-Nordammar-17(20)-en-21-oic acid, 16 β -hydroxy-3,6,7-trioxo-(?), methyl ester, acetate (preparation of)

RN 107277-22-5 HCAPLUS

CN 29-Nordammar-17(20)-en-21-oic acid, 16 β -hydroxy-3,6,7-trioxo-, methyl ester, acetate (7CI) (CA INDEX NAME)

Absolute stereochemistry.
Double bond geometry as shown.



L8 ANSWER 17 OF 23 HCAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 1961:9147 HCAPLUS [Full-text](#)

DOCUMENT NUMBER: 55:9147

ORIGINAL REFERENCE NO.: 55:1845a-c

TITLE: Storage iron in the animal body. I. Alterations in storage iron following experimental bleeding, liver damage, and iron administration

AUTHOR(S): Yoshioka, Yuzo

CORPORATE SOURCE: Univ. Hiroshima Med. School

SOURCE: Hiroshima Igaku (1959), 10, 2565-74

CODEN: HIRGAY; ISSN: 0367-5904

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

ED Entered STN: 22 Apr 2001

AB The changes in storage Fe in various organs of rabbits were studied under different conditions. In normal rabbits the ferritin (I) and hemosiderin (II) concns. were in the following order: spleen, liver, bone marrow, and kidney. The II content was higher than that of I in the liver, kidney, and bone marrow, while the reverse relation was observed in the spleen. Hemorrhagic anemia resulted in a marked decrease in storage Fe to a greater extent in spleen than in the liver, kidney, and bone marrow. In this case the predominant reduction was in I in the liver, spleen, and kidney, while II was more markedly reduced in the bone marrow. During the recovery phase after hemorrhage, a marked increase in liver II and a progressive decrease in I of all organs were observed. Recovery from the anemic state was much disturbed by the induction of liver damage, where a marked reduction of I and II occurred in the liver and spleen, and a slight decrease of I and a slight increase of II were seen in the kidney and bone marrow. Acceleration of recovery by Fe administration was accompanied by a marked increase in I and II in all organs. The increase was greatest in the liver.

IT 111663-25-3

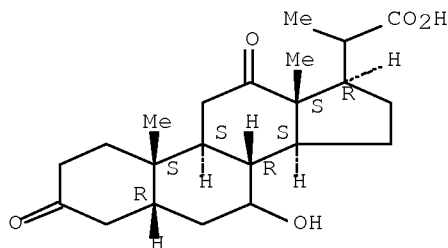
Serial No.:10/594,163

(Derived from data in the 6th Collective Formula Index (1957-1961))

RN 111663-25-3 HCAPLUS

CN 5 β -Pregnane-20-carboxylic acid, 7-hydroxy-3,12-dioxo- (6CI) (CA
INDEX NAME)

Absolute stereochemistry.



L8 ANSWER 18 OF 23 HCAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 1961:9146 HCAPLUS [Full-text](#)

DOCUMENT NUMBER: 55:9146

ORIGINAL REFERENCE NO.: 55:1844i,1845a

TITLE: The metabolism of bile acids. The metabolism of cholic acid and dehydrocholic acid by microorganisms
AUTHOR(S): Hoshita, Takahiko; Shimizu, Yoshimasa; Matsumura, Shinzo

CORPORATE SOURCE: Univ. Hiroshima Med. School
SOURCE: Hiroshima Igaku (1959), 10, 475-9
CODEN: HIRGAY; ISSN: 0367-5904

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

ED Entered STN: 22 Apr 2001

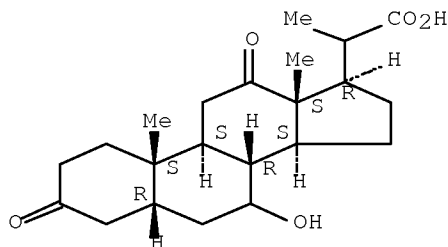
AB Cholate, when incubated with minced cat intestine, was metabolized to deoxycholate, while dehydrocholate was metabolized to 3,12-dioxo-7-hydroxybisanorcholanate.

IT 111663-25-3P, 5 β -Pregnane-20-carboxylic acid, 7-hydroxy-3,12-dioxo-
RL: PREP (Preparation)
(formation in dehydrocholic acid metabolism by intestinal bacteria)

RN 111663-25-3 HCAPLUS

CN 5 β -Pregnane-20-carboxylic acid, 7-hydroxy-3,12-dioxo- (6CI) (CA
INDEX NAME)

Absolute stereochemistry.



L8 ANSWER 19 OF 23 HCAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 1961:8771 HCAPLUS Full-text

DOCUMENT NUMBER: 55:8771

ORIGINAL REFERENCE NO.: 55:1785c-d

TITLE: Metabolism of cholic acid,
3 α ,12 α -dihydroxy-7-oxocholanic acid, and
dehydrocholic acid by microorganisms

AUTHOR(S): Shimizu, Yoshimasa

CORPORATE SOURCE: Univ. Hiroshima Med. School

SOURCE: Hiroshima Igaku (1958), 9, 2243-50

CODEN: HIRGAY; ISSN: 0367-5904

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

ED Entered STN: 22 Apr 2001

AB The incubation of cholic acid with fresh dog intestinal homogenates gave rise to 3 α ,12 α -dihydroxy-7-oxocholanic acid which was further metabolized to deoxycholic acid by one pathway and to dehydrobisanorcholic acid through dehydrocholic acid by another.

IT 102958-05-4P, 5 β -Pregnane-20-carboxylic acid, 3,7,12-trioxo-

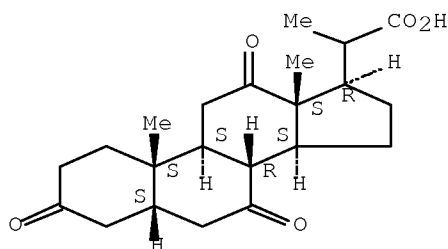
RL: PREP (Preparation)

(formation in cholic acid metabolism by intestinal microorganisms)

RN 102958-05-4 HCAPLUS

CN 5 β -Pregnane-20-carboxylic acid, 3,7,12-trioxo- (6CI) (CA INDEX NAME)

Absolute stereochemistry.



L8 ANSWER 20 OF 23 HCAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 1961:8769 HCAPLUS Full-text

DOCUMENT NUMBER: 55:8769

ORIGINAL REFERENCE NO.: 55:1785a

TITLE: Metabolism of bile acids [by *Aspergillus clavatus*]

AUTHOR(S): Kameo, Hitoshi

CORPORATE SOURCE: Univ. Hiroshima Med. School

SOURCE: Hiroshima Igaku (1958), 9, 1541-3

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

ED Entered STN: 22 Apr 2001

AB Dehydrocholate was metabolized to β -reductodehydrocholate and dehydrobisanorcholate by *A. clavatus* in a modified Czapek medium.

IT 102958-05-4P, 5 β -Pregnane-20-carboxylic acid, 3,7,12-trioxo-

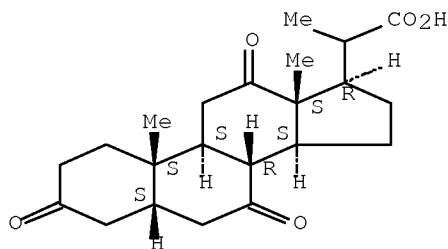
RL: PREP (Preparation)

(formation from dehydrocholic acid by *Aspergillus clavatus*)

RN 102958-05-4 HCAPLUS

CN 5 β -Pregnane-20-carboxylic acid, 3,7,12-trioxo- (6CI) (CA INDEX NAME)

Absolute stereochemistry.



L8 ANSWER 21 OF 23 HCAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 1956:1645 HCAPLUS Full-text

DOCUMENT NUMBER: 50:1645

ORIGINAL REFERENCE NO.: 50:388e-g

TITLE: Microbiological degradation of bile acid. III. Partial synthesis of methyl 7 α -acetoxy-3,12-dioxobisnorcholanate from cholic acid

AUTHOR(S): Hayakawa, Shohei

CORPORATE SOURCE: Okayama Univ. Med. School

SOURCE: Proceedings of the Japan Academy (1954), 30, 139-42

CODEN: PJACAW; ISSN: 0021-4280

DOCUMENT TYPE: Journal

LANGUAGE: English

ED Entered STN: 22 Apr 2001

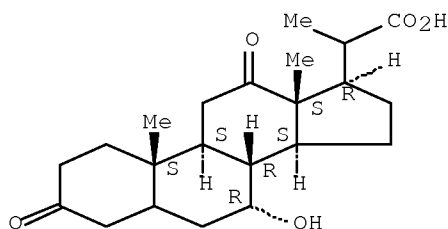
AB Me 3 α ,7 α -diacetoxy-12 α -hydroxybisnorcholanate (III), prepared from Me bisnorcholate by treatment with pyridine-Ac₂O in benzene, m. 180-2° (sintering at 110°). III was converted to Me 7 α -acetoxy-3 α ,12 α -dihydroxybisnorcholanate (IV), m. 142°, by treatment with HCl in MeOH at 25-30°, followed by dilution with H₂O, and recrystn. of the solids from benzene-petr. ether. IV with CrO₃ in AcOH yielded Me 7 α -acetoxy-3,12-dioxobisnorcholanate, m. 214-16°, giving a pos. Jaffe test, also prepared from the hydrogenated Me ester of I and Ac₂O. 7 α -Hydroxy-3,12-dioxobisnorcholanic acid, prepared from I by hydrogenation over Pd in EtOH, m. 249-52° and gave a positive Jaffe test; Me ester, prepared with CH₂N₂ in Et₂O, m. 162-4° (sintering at 157°) (from EtOAc-petr. ether, then C₆H₆-petr. ether).

IT 911672-84-9, 20-Pregnanecarboxylic acid, 7 α -hydroxy-3,12-dioxo- (and esters)

RN 911672-84-9 HCAPLUS

CN 20-Pregnanecarboxylic acid, 7 α -hydroxy-3,12-dioxo- (5CI) (CA INDEX NAME)

Absolute stereochemistry.



L8 ANSWER 22 OF 23 HCAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 1956:1644 HCAPLUS Full-text

DOCUMENT NUMBER: 50:1644

ORIGINAL REFERENCE NO.: 50:388b-e

TITLE: Microbiological degradation of bile acid. II.
Formation of 7-hydroxy-3,12-dioxobisnor-4,9(11)-choladienic acid from cholic acid by Actinomyces

AUTHOR(S): Hayakawa, Shohei

CORPORATE SOURCE: Okayama Univ. Med. School

SOURCE: Proceedings of the Japan Academy (1954), 30,
133-8

CODEN: PJACAW; ISSN: 0021-4280

DOCUMENT TYPE: Journal

LANGUAGE: English

ED Entered STN: 22 Apr 2001

AB Cholic acid-containing media (see above) (20 l.) fermented by Actinomyces number 1164 was concentrated (after filtration to remove the microbial cells), the concentrate acidified with HCl, and chilled. After 20 hrs. at 0°, crystals were observed. These were recrystd. from MeOH (yield of 4 g. from 60 g. of cholic acid added to medium), and the dioxime, prepared from the Me ester of this acid (see previous paper), analyzed 5.97% N (oxime of 7-hydroxy-3,12-dioxobisnor-4,9(11)-choladienate analyzed 5.95% N). Hydrogenation of the Me ester gave needles (m. 166-9°) which gave a pos. Jaffe test and neg. Shimizu-Mizuhara test, and was tentatively identified as Me 7-hydroxy-3,12-dioxobisnorcholatanate (II). The dioxime of II was prepared and m: 232-4°, N content, 6.53%. Me dehydrobisnorcholate, prepared from II by treatment with CrO3, m. 197-9°, identical with that of a synthetic sample; trioxime, m. 267-9°, N content, 9.57%, values similar to those obtained with synthetic materials. The ultraviolet spectra of the parent compound showed an absorption maximum at 240.3 mμ (ε = 11.15 + 104), and of the Me ester, 242.1 mμ (ε = 11.98 + 104). The data collected support the view that I is 7-hydroxy-3,12-dioxobisnor-4,9(11)-choladienic acid.

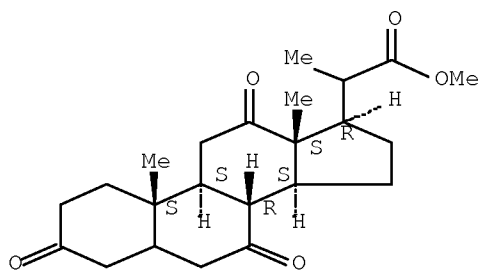
IT 881385-35-9F, 20-Pregnanecarboxylic acid, 3,7,12-trioxo-, methyl ester 911495-03-9F, 20-Pregnanecarboxylic acid, 7-hydroxy-3,12-dioxo-, methyl ester

RL: PREP (Preparation)
(preparation of)

RN 881385-35-9 HCAPLUS

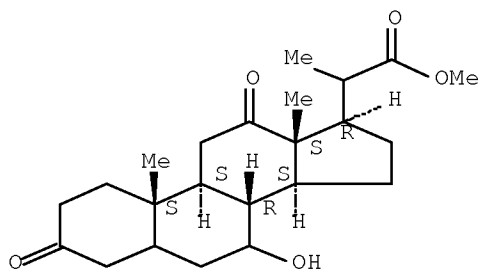
CN 20-Pregnanecarboxylic acid, 3,7,12-trioxo-, methyl ester (5CI) (CA INDEX NAME)

Relative stereochemistry.



RN 911495-03-9 HCAPLUS
 CN 20-Pregnanecarboxylic acid, 7-hydroxy-3,12-dioxo-, methyl ester (5CI) (CA INDEX NAME)

Absolute stereochemistry.



L8 ANSWER 23 OF 23 HCAPLUS COPYRIGHT 2009 ACS on STN
 ACCESSION NUMBER: 1956:1643 HCAPLUS [Full-text](#)
 DOCUMENT NUMBER: 50:1643
 ORIGINAL REFERENCE NO.: 50:387h-i,388a-c
 TITLE: Microbiological degradation of bile acid. I. On
 β -oxidation and unsaturation of cholic acid by
 Actinomyces
 AUTHOR(S): Hayakawa, Shohei
 CORPORATE SOURCE: Okayama Univ. Med. School
 SOURCE: Proceedings of the Japan Academy (1954), 30,
 128-32
 CODEN: PJACAW; ISSN: 0021-4280
 DOCUMENT TYPE: Journal
 LANGUAGE: English
 ED Entered STN: 22 Apr 2001
 AB When Actinomyces number 1164 (Okayama Tobacco Laboratory, Japan Monopoly
 Bureau) was grown for 14 days (incubation temperature 30°) on a medium
 containing 6 g. cholic acid, 2 g. (NH₄)₂SO₄, 2 g. K₂HPO₄, 1 g. MgSO₄·7H₂O,
 0.02 g. FeCl₃·6H₂O, and 2 l. H₂O (pH adjusted to 7.2 with NaOH) in culture
 without agitation (100 ml. medium/500 ml. conical flask) the pH dropped to
 5.8, a neg. Pettenkofer test was obtained, and no precipitate was observed
 when the pH was lowered with HCl. The cell-free liquid was concentrated (at
 pH 7.2) in vacuo to about 0.1 volume, and 2 substances crystallized An acid
 (I), m. 280-2° (decomposition), gave neg. results to the Hammarsten-Yamasaki,
 the Mylius, and Shimizu-Mizuhara tests. A yellow Liebermann test was
 obtained, while pos. Jaffe and Zimmermann tests were obtained only after 5

Serial No.:10/594,163

min. I was slightly soluble in H₂O, MeOH, EtOH, and acetone, and insol. in Et₂O, petr. ether, and benzene. Mol. weight from titration was 377.25; analysis for C₂₂H₂₈O₅: C, 70.9; H, 7.58; found, C, 70.1; H, 8.01. The Me ester, prepared by treatment with CH₂N₂ in Et₂O, m. 237° (sintering) (from MeOH). I was hydrogenated over Pt oxide, but oils were obtained. Oxidation of the Me ester with CrO₃ in AcOH gave an ester identified as Me bisnorcholelate by comparison with a sample prepared from cholic acid by the procedure of Morsman, et al. (C.A. 31, 3062.4). A second acid from the fermentation C₂₀-24H₂₈-34O₅-6, m. 248° (decomposition), and had the same qual. tests and solubilities as the other acid.

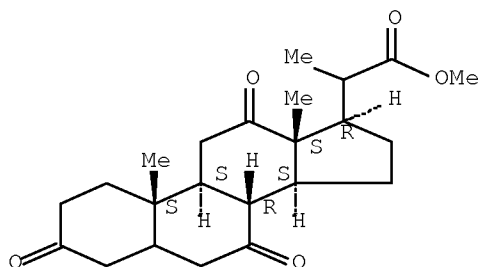
IT 881385-35-9P, 20-Pregnanecarboxylic acid, 3,7,12-trioxo-, methyl ester

RL: PREP (Preparation)
(preparation of)

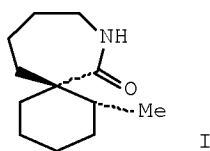
RN 881385-35-9 HCAPLUS

CN 20-Pregnanecarboxylic acid, 3,7,12-trioxo-, methyl ester (5CI) (CA INDEX NAME)

Relative stereochemistry.



L26 ANSWER 1 OF 8 HCAPLUS COPYRIGHT 2009 ACS on STN
 ACCESSION NUMBER: 2009:61598 HCAPLUS Full-text
 DOCUMENT NUMBER: 150:214146
 TITLE: Synthesis of Enantiopure Bicyclic
 α,α -Disubstituted Spirolactams via
 Asymmetric Birch Reductive Alkylation
 AUTHOR(S): Gueret, Stephanie M.; O'Connor, Patrick D.; Brimble,
 Margaret A.
 CORPORATE SOURCE: Department of Chemistry, University of Auckland,
 Auckland, N. Z.
 SOURCE: Organic Letters (2009), 11(4), 963-966
 CODEN: ORLEF7; ISSN: 1523-7060
 PUBLISHER: American Chemical Society
 DOCUMENT TYPE: Journal
 LANGUAGE: English
 ED Entered STN: 16 Jan 2009
 GI

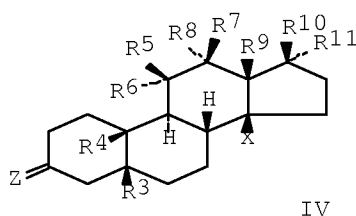
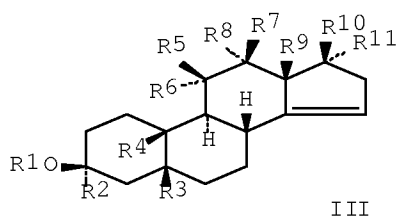
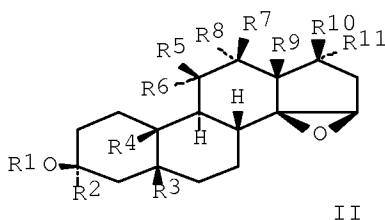
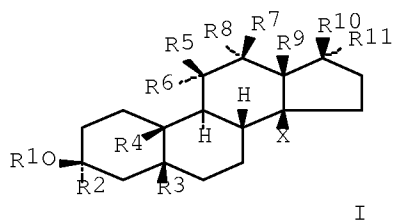


AB The synthesis of enantiopure bicyclic α,α -disubstituted spirolactams is described using a diastereoselective Birch reductive alkylation as the key step. Hydrogenation of the resultant alkylated cyclohexadienes followed by intramol. cyclization provides access to enantiopure 8-azaspiro[5.6]dodecan-7-ones, e.g. I.

REFERENCE COUNT: 37 THERE ARE 37 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L26 ANSWER 2 OF 8 HCAPLUS COPYRIGHT 2009 ACS on STN
 ACCESSION NUMBER: 2005:100362 HCAPLUS Full-text
 DOCUMENT NUMBER: 143:44004
 TITLE: Total synthesis of 14 beta-fluorosteroids via the
 transannular Diels-Alder reaction
 INVENTOR(S): Beaubien, Sylvie; Deslongchamps, Pierre
 PATENT ASSIGNEE(S): Neokimia Inc., Can.
 SOURCE: Can. Pat. Appl., 321 pp.
 CODEN: CPXXEB
 DOCUMENT TYPE: Patent
 LANGUAGE: French
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
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CA 2418458	A1	20040806	CA 2003-2418458	20030206
PRIORITY APPLN. INFO.:			CA 2003-2418458	20030206
OTHER SOURCE(S):	MARPAT	143:44004		
ED Entered STN:		04 Feb 2005		
GI				



AB A series of novel digitalis steroid analogs, including derivs. containing a 14 β -fluoro substituent, I [R1 = H, Me, C2-6-alkyl (optionally substituted with NRbRc or NHC(:NRd)NRbRc], (un)substituted (CH2)nPh, C(:O)Ra, mono-, di-, trisaccharyl, aminoacyl, di-, tripeptidyl; R2 = H, Me, OR1; R3 = H, Me, ORd, OC(:O)Rd; R4 = H, Me, OC(:O)Rd, CHO, (CH2)mORd, (CH2)nNRbRc; R5, R6, R7, R8 = H, ORd, OC(:O)Rd; R9 = H, Me; R10 = H, (CH:CR9)p(CH2)m(CHR9)nA, (CH:CR9)p(CHR12)(CHR9)nA, (CHR9)m(CH:CR9)pB, furyl derivative, furanone derivative, pyranone derivative, pyrazin-4-yl; R11 = H, ORa, OC(:O)Ra; R12 = ORg, NRhRi; NHC(:Y)NRhRi; Rg = Ra; Rh, Ri = Ra with the proviso that, when Rh = H, Ri = NHC(:NH)NH2; Y = O, S, NRA; Ra = H, Me, C2-6-alkyl (optionally substituted with NRbRc or NHC(:NRd)NRbRc), (un)substituted (CH2)nPh; Rb, Rc = H, Me, C2-6-alkyl, (un)substituted (CH2)nPh; NRbRc = 5- or 6-membered monoheterocyclic ring, optionally containing N or O; Rd = H, Me, C2-6-alkyl, (un)substituted (CH2)nPh; X = F, Cl, Br, I; A = NO2, CO2Ra, CHO, R12, CR9:NR12; B = NO2, CO2Ra, CHO, CR9:NR12; m = 0-6; n = 1-6; p = 0-3), II, III and IV [Z = O, S, NR12], their tautomers, R/S-enantiomers (excluding the steroid nucleus), or E/Z double bonds isomers, have been synthesized. I-IV have been shown to display binding affinity for the ouabain receptor as well as activity in the inhibitions of Na⁺,K⁺-ATPase. These compds. have implications in therapy for a number of medical indications, most notably congestive heart failure, hypertension and cancer. In addition, these mols. can function as natriuretic/diuretic agents and neuromodulators. Multiple methods for the synthesis of these compds. are also presented.

L26 ANSWER 3 OF 8 HCAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 1999:583184 HCAPLUS Full-text

DOCUMENT NUMBER: 131:199937

TITLE: Preparation of deoxymannojirimycin from

(4R)-3-benzyl-4-(methoxycarbonyl)-2-oxazolidinone

INVENTOR(S): Katsumura, Shigeo; Asano, Hiroshi; Murakami, Masanobu;
Iwama, Seiji

PATENT ASSIGNEE(S): Daiso Co., Ltd., Japan

Serial No.:10/594,163

SOURCE: Jpn. Kokai Tokkyo Koho, 8 pp.
 CODEN: JKXXAF
 DOCUMENT TYPE: Patent
 LANGUAGE: Japanese
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 11246524	A	19990914	JP 1998-43381	19980225
JP 3317233	B2	20020826		

PRIORITY APPLN. INFO.: JP 1998-43381 19980225
 OTHER SOURCE(S): CASREACT 131:199937; MARPAT 131:199937
 ED Entered STN: 16 Sep 1999
 GI

* STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY - AVAILABLE VIA OFFLINE PRINT *

AB Deoxymannojirimycin, a known anticancer and antiviral agent, is prepared from (4R)-3-benzyl-4-(methoxycarbonyl)-2-oxazolidinone (I; R = OMe) without using expensive and difficult-to-handle reagents in a highly stereoselective method. The process involved treatment of O-(~~tert-butyl~~~~dimethylsilyl~~)propargyl alc. with BuLi in THF/hexamethylphosphoramide at -78° for 1 h and coupling with (4R)-3-benzyl-4-(methoxycarbonyl)-2-oxazolidinone (I; R = OMe) at -100° for 15 min to give propynyl ketone (I; R = ~~tert~~-BuMe₂SiOCH₂C.tplbond.C) (65.5%), reduction of the latter ketone with diisobutylaluminum-2,6-di-~~tert~~-butyl-4-methylphenoxide in toluene at 0° for 10 min to 4-(1,4-dihydroxy-2-butenyl)oxazolidinone (II) (92.2%), hydrogenation of the latter compound over Lindlar catalyst to N-benzyl-4-(cis-1,4-dihydroxy-2-butenyl)oxazolidinone (III; R₁ = CH₂Ph, R₂ = H, R₃ = ~~tert~~-BuMe₂Si) (100%), ~~Birch~~ reduction of the N-benzyl-4-(cis-1,4-dihydroxy-2-butenyl)oxazolidinone with Na in NH₃(l) at -78° to give 4-(cis-1,4-dihydroxy-2-butenyl)oxazolidinone (III; R₁ = R₂ = H, R₃ = ~~tert~~-BuMe₂Si) (79.9%), silylation of the latter alc. with ~~tert-butyl~~~~dimethylsilyl~~ chloride in the presence of imidazole in DMF (96.4%) and selective desilylation with a mixture of 33% aqueous HF and MeCN at -20° for 30 min to give III (R₁ = R₃ = H, R₂ = ~~tert~~-BuMe₂Si) (97.6%). Mesylation of the latter compound with methanesulfonyl chloride 4-dimethylaminopyridine and Et₃N in DMF followed by treatment of the crude mesylate (III; R₁ = H, R₂ = ~~tert~~-BuMe₂Si, R₃ = SO₂Me) with NaH in DMF at 0° for 1 h gave a bicyclic oxazolidinone derivative (IV) (79.9%) which underwent OsO₄-catalyzed dihydroxylation with N-methylmorpholine-N-oxide in a mixture of ~~tert~~-Bu alc. and H₂O at 0° to a diol (V; R₄ = H) (85.6%). Acetonation of the latter diol with 2,2-dimethoxypropane in acetone in the presence of pyridinium p-toluenesulfonate at room temperature for 20 h gave an acetonide (V; R₄R₄ = Me₂C) (92.1%) which was refluxed with a mixture of 6 N aqueous NaOH and dioxane for 2 days to 70.7% give deoxymannojirimycin acetonide (VI). Deacetonation was effected by refluxing the latter acetonide in MeOH in the presence of concentrated H₂SO₄ followed by purification on an ion exchange column (I-X4, OH- form) to give 76.5% deoxymannojirimycin.

L26 ANSWER 4 OF 8 HCAPLUS COPYRIGHT 2009 ACS on STN
 ACCESSION NUMBER: 1999:512071 HCAPLUS Full-text
 DOCUMENT NUMBER: 140:253359

TITLE: An efficient synthesis of pironetins employing a useful chiral building block, (1S,5S,6R)-5-hydroxybicyclo[4.1.0]heptan-2-one
 AUTHOR(S): Watanabe, Hidenori; Watanabe, Hiroyuki; Bando,

Serial No.:10/594,163

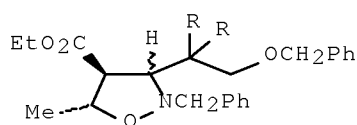
CORPORATE SOURCE: Masahiko; Kido, Masaru; Kitahara, Takeshi
 Department of Applied Biological Chemistry, Graduate
 School of Agricultural and Life Sciences, The
 University of Tokyo, Tokyo, 113-8657, Japan
 SOURCE: Tetrahedron (1999), 55(32), 9755-9776
 CODEN: TETRAB; ISSN: 0040-4020
 PUBLISHER: Elsevier Science Ltd.
 DOCUMENT TYPE: Journal
 LANGUAGE: English
 OTHER SOURCE(S): CASREACT 140:253359
 ED Entered STN: 18 Aug 1999
 GI

* STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY - AVAILABLE VIA OFFLINE PRINT *

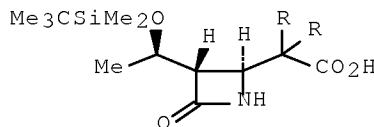
AB A convergent total synthesis of pironetin (I; R = Me) and related compound I (R = H) using a chiral building block, (1S,5S,6R)-5-hydroxybicyclo[4.1.0]heptan-2-one (II) is described. Both the dithiane III and the epoxide IV with proper substituents were employed as coupling partners to construct the whole carbon skeleton V, which was converted to (-)-pironetin (I; R = Me) and (-)-I (R = H) in few steps. The usefulness of II for polyketide synthesis was demonstrated.

REFERENCE COUNT: 44 THERE ARE 44 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L26 ANSWER 5 OF 8 HCAPLUS COPYRIGHT 2009 ACS on STN
 ACCESSION NUMBER: 1996:299594 HCAPLUS Full-text
 DOCUMENT NUMBER: 125:58151
 ORIGINAL REFERENCE NO.: 125:11177a,11180a
 TITLE: Synthesis of 3-[1-(tert-butyldimethylsilyloxy)ethyl]-4-carboxymethyl-2-azetidinone derivatives
 AUTHOR(S): Seo, Min Hyo; Lee, Youn Young; Goo, Yang Mo
 CORPORATE SOURCE: Department Chemistry, Seoul National University, Seoul, 151-742, S. Korea
 SOURCE: Bulletin of the Korean Chemical Society (1996), 17(4), 314-321
 CODEN: BKCSDE; ISSN: 0253-2964
 PUBLISHER: Korean Chemical Society
 DOCUMENT TYPE: Journal
 LANGUAGE: English
 OTHER SOURCE(S): CASREACT 125:58151
 ED Entered STN: 21 May 1996
 GI



I



II

AB Isoxazolidine derivs. I (R = H, Me) were synthesized from N-benzyl-C-(2-benzyloxyethyl)nitrones by 1,3-dipolar cycloaddn. with (E)-Et crotonate. The

isoxazolidine derivs. were converted to β -amino acid esters by reduction with zinc in acetic acid. The β -amino acid esters were reacted with methylmagnesium bromide to give the 2-azetidinones. The benzyl group of 2-azetidinones were removed by Birch reduction. The products were oxidized with PDC to give 3-[1-(t-butyldimethylsilyloxy)ethyl]-4-carboxymethyl-2-azetidinone derivs II (R = H, Me).

L26 ANSWER 6 OF 8 HCAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 1993:603806 HCAPLUS Full-text
 DOCUMENT NUMBER: 119:203806
 ORIGINAL REFERENCE NO.: 119:36373a,36376a
 TITLE: A novel constrained reduced-amide inhibitor of HIV-1 protease derived from the sequential incorporation of γ -turn mimetics into a model substrate
 AUTHOR(S): Newlander, Kenneth A.; Callahan, James F.; Moore, Michael L.; Tomaszek, Thaddeus A., Jr.; Huffman, William F.
 CORPORATE SOURCE: Dep. Med. Chem., SmithKline Beecham Pharm., King of Prussia, PA, 19406, USA
 SOURCE: Journal of Medicinal Chemistry (1993), 36(16), 2321-31
 CODEN: JMCMAR; ISSN: 0022-2623
 DOCUMENT TYPE: Journal
 LANGUAGE: English
 OTHER SOURCE(S): CASREACT 119:203806
 ED Entered STN: 13 Nov 1993
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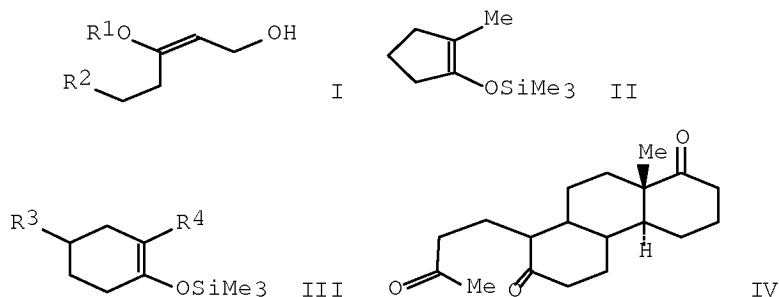
* STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY - AVAILABLE VIA OFFLINE PRINT *

AB C7 mimetics, designed to lock three amino acid residues of a peptide chain into a γ -turn conformation, were introduced sequentially between the P3 to P2' positions of a model HIV-1 protease substrate I resulting in compds. I, II and III as probes for conformational requirements in binding to HIV-1 protease. The above I-III were obtained as 2 diastereoisomers. A diastereoisomer of II with the C7 mimetic replacing Asn-Tyr-Pro, corresponding to the P2 through P1' positions of substrate, was found to be an inhibitor with a K_i of 147 μ M. Reduction of the amide bond in the C7 mimetic of the above diastereoisomer of II resulted in a novel constrained reduced-amide mimetic IV with a K_i of 430 nM. This corresponds to over a 300-fold improvement in inhibitory activity over the original C7 mimetic. The inhibitory activity of mimetic IV was in addition found to be 44-fold better than a similar linear reduced-amide containing inhibitor H-Ser-Ala-Ala-Phe ψ [CH₂N]Pro-Val-Val-NH₂. The synthesis of these mimetics are described.

L26 ANSWER 7 OF 8 HCAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 1990:119219 HCAPLUS Full-text
 DOCUMENT NUMBER: 112:119219
 ORIGINAL REFERENCE NO.: 112:20211a,20214a
 TITLE: A new bis-annulation method. Application to steroid synthesis
 AUTHOR(S): Poirier, Jean Marie; Hennequin, Laurent
 CORPORATE SOURCE: Lab. Chim. Org., Fac. Sci. Tech. Rouen, Mont Saint Aignan, 76134, Fr.
 SOURCE: Tetrahedron (1989), 45(13), 4191-202

DOCUMENT TYPE: Journal
 LANGUAGE: English
 OTHER SOURCE(S): CASREACT 112:119219
 ED Entered STN: 31 Mar 1990
 GI



AB New bis-annulation reagents I [R1 = ~~tert~~-butyldimethylsilyl, R2 = (2-methyl-2-dioxolanyl)methyl, MeC(:CH₂)CH₂; R1 = Me, R2 = CH₂:CHCH₂] and R2CH₂CH₂COCH:CH₂ (R2 = same) are described. With monocyclic enolates equivalent II and III (R2 = H, R3 = Me; R2 = CMe₃, R3 = H), these reagents led to the formation of tricyclic compds. in few steps. The utility of the method is tested for the steroid rings synthesis. Thus, triketone IV was prepared

L26 ANSWER 8 OF 8 HCAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 1987:195930 HCAPLUS Full-text

DOCUMENT NUMBER: 106:195930

ORIGINAL REFERENCE NO.: 106:31748h,31749a

TITLE: Improved methods for the reductive alkylation of methoxybenzoic acids and esters. Applications to the synthesis of bicyclic ketones

AUTHOR(S): Hamilton, Robert J.; Mander, Lewis N.; Sethi, S. Paul

CORPORATE SOURCE: Res. Sch. Chem., Aust. Natl. Univ., Canberra, 2601, Australia

SOURCE: Tetrahedron (1986), 42(11), 2881-92

CODEN: TETRAB; ISSN: 0040-4020

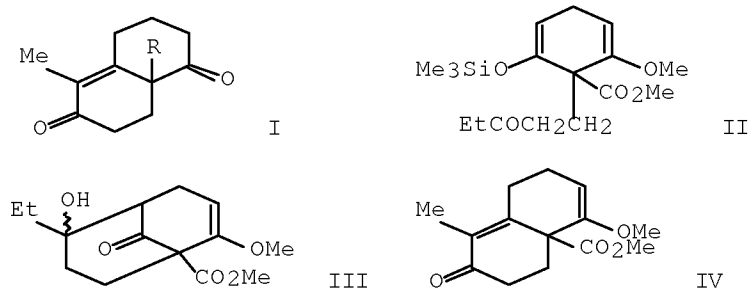
DOCUMENT TYPE: Journal

LANGUAGE: English

OTHER SOURCE(S): CASREACT 106:195930

ED Entered STN: 13 Jun 1987

GI



AB A series of methoxybenzoic acids and esters was reduced by metal-NH₃ solns. and the resulting 1,4-dihydro products were either alkylated in situ or isolated and alkylated subsequently. Three different types of alkyl iodides were employed to introduce the elements of a butanone or pentanone side-chain as a prelude to adding a fused six-membered ring, thereby completing the preparation of several analogs of the Wieland-Miescher ketone I (R = H), in which the angular substituent was oxygenated. Thus, 2,6-MeO(Me₃SiO)C₆H₃CO₂Me was treated sequentially with K-Me₃COH in NH₃, LiBr, and then ICH₂CH₂CH(OSiMe₃)CH₂Me, followed by Me₂SO and DCC to give 60% the cyclohexadienecarboxylate II. Treating II with Bu₄NF in THF gave up to 90% diastereomeric bicyclononenecarboxylates III, which rearranged on treatment with K₂CO₃ in MeOH to give 75% bicyclodecadienecarboxylate IV. Demethylation of IV with Hg(NO₂)₂ in MeCN-H₂O gave 91% I (R = CO₂Me).

SEARCH HISTORY

FILE 'CASREACT' ENTERED AT 14:16:38 ON 10 MAR 2009

L1 STRUCTURE UPLOADED
 L2 0 SEA SSS SAM L1 (0 REACTIONS)
 L3 1 SEA SSS FUL L1 (3 REACTIONS)

FILE 'REGISTRY' ENTERED AT 14:23:55 ON 10 MAR 2009

L4 STRUCTURE UPLOADED
 L5 0 SEA SSS SAM L4
 L6 22 SEA SSS FUL L4

FILE 'HCAPLUS' ENTERED AT 14:29:17 ON 10 MAR 2009

L7 23 SEA SPE=ON ABB=ON PLU=ON L6
 L8 23 SEA SPE=ON ABB=ON PLU=ON L7 AND (PRY<=2005 OR AY<=2005 OR
 PY<=2005)
 L9 25 SEA SPE=ON ABB=ON PLU=ON KOYAKUMARU K?/AU
 L10 227 SEA SPE=ON ABB=ON PLU=ON SUGIOKA T?/AU
 L11 10 SEA SPE=ON ABB=ON PLU=ON OHZONO S?/AU
 L12 1278 SEA SPE=ON ABB=ON PLU=ON NAKAGAWA N?/AU
 L13 7 SEA SPE=ON ABB=ON PLU=ON (L9 OR L10 OR L11 OR L12) AND L8
 L14 2 SEA SPE=ON ABB=ON PLU=ON L9 AND L10 AND L11 AND L12
 L15 3 SEA SPE=ON ABB=ON PLU=ON L12 AND L9
 L16 2 SEA SPE=ON ABB=ON PLU=ON L12 AND L10
 L17 2 SEA SPE=ON ABB=ON PLU=ON L12 AND L11
 L18 3 SEA SPE=ON ABB=ON PLU=ON (L14 OR L15 OR L16 OR L17)
 L19 1485 SEA SPE=ON ABB=ON PLU=ON BIRCH REDUCTION/CT OR BIRCH
 REDUCTION/BI
 L20 0 SEA SPE=ON ABB=ON PLU=ON L8 AND L19
 L21 0 SEA SPE=ON ABB=ON PLU=ON L19 AND (L9 OR L10 OR L11 OR L12)
 L22 575 SEA SPE=ON ABB=ON PLU=ON METAL LITHIUM/BI
 L23 0 SEA SPE=ON ABB=ON PLU=ON L19 AND L22
 L24 0 SEA SPE=ON ABB=ON PLU=ON L22 AND (L9 OR L10 OR L11 OR L12)
 L25 7329 SEA SPE=ON ABB=ON PLU=ON TERT BUTYLDIMETHYLSILYL/BI OR
 T-BUTYLDIMETHYLSILYL/BI
 L26 8 SEA SPE=ON ABB=ON PLU=ON L19 AND L25